Matthiessen & Hegeler Zinc Company Site
Operable Unit 2
LaSalle, Illinois

Phase II Sampling and Analysis Plan

SulTRAC

May 30, 2008

Ms. Demaree Collier
Work Assignment Manager (SR-6J)
Remedial Response Unit No. 1
U.S. Environmental Protection Agency Region 5
77 West Jackson Boulevard
Chicago, IL 60604

Subject: **Matthiessen and Hegeler Zinc Company Site, Operable Unit 2**
LaSalle, Illinois
Phase II Sampling and Analysis Plan for Remedial Investigation/ Feasibility Study
Response Action Contract (RAC) 2 EP-S5-06-02
Work Assignment No. 032-RICO-B568

Dear Ms. Collier:

In accordance with the work plan approved by the U.S. Environmental Protection Agency on April 30, 2008, SulTRAC is submitting this Sampling and Analysis Plan (SAP) for your review. This SAP includes the Field Sampling Plan and Quality Assurance Project Plan as attachments for the Matthiessen and Hegeler Zinc Company Site, Operable Unit 2, in LaSalle, Illinois.

If you have any questions regarding the site-specific plans please contact me at (312) 443-0550, extension 16, or email jknoepfle@onesullivan.com.

Sincerely,

Jennifer Knoepfle
SulTRAC Project Manager

Enclosure

cc: Parveen Vij, US EPA Project Officer (letter only)
Ron Riesing, SulTRAC Program Manager (letter only)

File

REMEDIAL ACTION CONTRACT 2 FOR
REMEDIAL, ENFORCEMENT OVERSIGHT, AND
NON-TIME CRITICAL REMOVAL ACTIVITIES
IN REGION 5

PHASE II SAMPLING AND ANALYSIS PLAN
MATTHIESSEN AND HEGELER ZINC COMPANY SITE
OPERABLE UNIT 2
LASALLE COUNTY, LASALLE, ILLINOIS

Prepared for
United States Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604

Date Submitted:	May 30, 2008
EPA Region:	5
Work Assignment No:	032-RICO-B568
Contract No:	EP-S5-06-02
Prepared by:	SulTRAC
Project Manager:	Jennifer Knoepfle
Telephone No:	(312) 443-0550 ext.16
EPA Work Assignment Manager:	Demaree Collier
Telephone No:	(312) 886-0214

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1.0 INTRODUCTION

SulTRAC has prepared this sampling and analysis plan (SAP) to meet the requirements specified in the statement of work (SOW) for the Matthiessen and Hegeler Site (M&H Site), operable unit 2 (OU2), located in La Salle County, La Salle, Illinois, under the U.S. Environmental Protection Agency (EPA) Resmedial Action Contract (RAC) II for Region 5, Contract No.EP-S5-06-02, Work Assignment (WA) No. 032-RICO-B568. This SAP describes sampling and analysis activities to be conducted by SulTRAC during the Phase II remedial investigation/feasibility study (RI/FS) at the M&H Site. The SAP consists of the field sampling plan (FSP) (Attachment A) and the quality assurance project plan (QAPP) (Attachment B); it is among the site-specific plans to be prepared under the WA in accordance with Task 1 of the EPA SOW dated February 28, 2008, for the M&H Site. Quality assurance (QA) and quality control (QC) protocols associated with the sampling and analysis activities are presented in the QAPP.

REMEDIAL ACTION CONTRACT 2
FOR REMEDIAL, ENFORCEMENT OVERSIGHT, AND
NON-TIME CRITICAL REMOVAL ACTIVITIES
IN REGION 5

ATTACHMENT A

PHASE II FIELD SAMPLING PLAN
MATTHIESSEN AND HEGELER ZINC COMPANY SITE
OPERABLE UNIT 2
LASALLE COUNTY, ILLINOIS

Prepared for
United States Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604

Date Submitted:	August 14, 2008
EPA Region:	5
Work Assignment No:	032-RICO-B568
Contract No:	EP-S5-06-02
Prepared by:	SulTRAC
Project Manager:	Jennifer Knoepfle
Telephone No:	(312) 443-0550, ext.16
EPA Work Assignment Manager:	Demaree Collier
Telephone No:	(312) 886-0214

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	NO. 3, FEBRUARY 2007
SOP In-vitro	THE IN-VITRO METHOD, RELATIVE BIOAVAILABILITY LEACHING PROCEDURE, 2003
SOP PCB	EPA Method 9078: SCREENING TEST METHOD FOR POLYCHLORINATED BIPHENYLS IN SOILS

ACRONYMS AND ABBREVIATIONS

µm	Micrometer
AERMOD	Air dispersion modeling
AST	Aboveground storage tank
ATV	All-terrain vehicle
BAF	Bioaccumulation factor
bgs	Below ground surface
BERA	Baseline ecological risk assessment
°C	Degrees Celsius
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COC	Chain of custody
CRL	Central Regional Laboratory
DOT	Department of Transportation
DPT	Direct-push technology
EPA	United States Environmental Protection Agency
FIELDS	Field Environmental Decision Support Team
FS	Feasibility study
FSP	Field sampling plan
FTL	Field team leader
HASP	Health and safety plan
HDPE	High-density polyethylene
HHRA	Human health risk assessment
ICRR	Illinois Central Rail Road
ID	Identity
IDW	Investigation-derived waste
IEPA	Illinois Environmental Protection Agency
IDPH	Illinois Department of Public Health
LEGS	Laboratory for Environmental and Geological Studies
LIMS	Laboratory information management system
MA	Modified Analysis
M&H	Matthiessen and Hegeler Zinc
MCE	Mixed cellulose ester
mm	Millimeter
MS	Matrix spike
MSD	Matrix spike duplicate

NA	Not applicable
NaOH	Sodium hydroxide
NIOSH	National Institute for Occupational Safety and Health
NPL	National Priorities List
NR	Not required
OU	Operable unit
PCB	Polychlorinated biphenyl
PID	Photoionization detector
PPE	Personal protective equipment
PRP	Potentially responsible party
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl chloride
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
RAC	Remedial action contract
RAT	Rapid assessment tool
RBLP	Relative bioavailability leaching procedure
RI	Remedial investigation
ROD	Record of Decision
ROW	Right of way
RR	Railroad
SAP	Sampling and analysis plan
SARA	Superfund Amendments and Reauthorization Act
SLERA	Screening-level ecological risk assessment
SMO	Sample Management Office
SPLP	Synthetic precipitation leaching procedure
SOP	Standard operating procedure
SOW	Statement of work
SVOC	Semivolatile organic compound
TBD	To be determined
TCE	Trichloroethene
TCLP	Toxicity characteristic leaching procedure
TOC	Top of casing
VOC	Volatile organic compounds
WA	Work assignment
WAM	Work assignment manager

XRF	X-ray fluorescence

1.0 INTRODUCTION

SulTRAC has prepared this Phase II field sampling plan (FSP) as part of the sampling and analysis plan (SAP) for the Matthiessen and Hegeler Zinc Company Site (M&H Site) in LaSalle, LaSalle County, Illinois, under the United States Environmental Protection Agency (EPA) Remedial Action Contract (RAC) II for Region 5, Contract No. EP-S5-06-02, Work Assignment (WA) No. 032-RICO-B568. The M&H Site is being addressed under three separate remedial investigation/feasibility study (RI/FS) WAs, as the site is divided into two operable units (OU), OU1 and OU2. SulTRAC is currently providing technical support for both the potentially responsible party (PRP)-lead RI/FS (WA 015-RSBD-B568), OU1, and this fund-lead RI/FS, OU2. Phase I of the fund-lead RI/FS was conducted under WA 016-RICO-B568 and is scheduled for close-out in December 2008. Phase II of the RI/FS activities is being conducted under this work assignment, WA 032-RICO-B568. The SAP consists of this FSP (Attachment A) and the quality assurance project plan (QAPP) (Attachment B), which are among the site-specific plans to be prepared under the WA in accordance with Task 1 of the EPA statement of work (SOW) (EPA 2008). Quality assurance (QA) and quality control (QC) protocols, associated with sampling and analysis activities, are presented in the QAPP.

This FSP describes sampling activities to be performed by SulTRAC during the Phase II RI/FS at the M&H Site and is specific to OU2. The scope of the FSP, as outlined in the M&H Site work plan for OU2 (SulTRAC 2008a), has been developed by interacting with the EPA, synthesizing Phase I analytical and field survey results, conducting an April 2008 site visit during a time when site vegetation was minimal, enhancing views, and reviewing both regulatory and industrial related site documents.

Based on the original limited site information pertaining to contamination, SulTRAC proposed, and the EPA concurred (EPA 2007a), that the OU2 M&H Site RI/FS activities should be divided into two phases. Phase I was addressed under WA No. 016-RICO-B568 and included investigation activities focused on characterizing the nature of M&H Site contamination in a biased sampling approach. Phase II investigation activities described in this FSP focus on delineating the extent of contamination. Another objective of the Phase II FSP is to address any contamination characterization data gaps from Phase I. The Phase I investigation was completed during the summer and fall of 2007; therefore, this FSP will detail only Phase II field activities. Following receipt of data from the Phase II investigation, an RI report will be prepared containing the results of the Phase I and Phase II investigations.

2.0 SITE DESCRIPTION AND HISTORY

The entire M&H Site, located in LaSalle, LaSalle County, Illinois, encompasses approximately 160 acres inclusive of inactive primary zinc smelting operations and associated abandoned and partially demolished buildings, a rolling mill, and the active Carus Chemical Company and its property (see Figure 1). The M&H Site is bounded by the Little Vermilion River to the north and east and by private residences to the south and west. Tracts of farmland and a limestone quarry are located across the Little Vermilion River to the north and east of the site, respectively. The M&H Site is defined by an upland area over much of the western and northern portions of the site, a 6-acre slag pile on the eastern side, and a retention pond to the south. The upland areas are flat and encompass both the OU1 current processing area and the OU2 former processing area. The 6-acre slag pile is located to the east of the main plant area in OU1 and runs the length of the OU1 property. The slag pile is just west of the river, and portions extend into the Little Vermilion River (see Figure 1). This slag pile is the topographic high of the M&H Site with an elevation between 40 and 120 feet higher than the river surface. An abandoned sewer line, as well as three drainage outfall pipes located on the northeast portion of the OU2 property, all run east-west across the property and serve as a transport mechanism for surface water runoff directly into the Little Vermilion River. A wetland is located approximately 0.5 mile upstream from the M&H Site, and the Illinois River is located approximately 1 mile downstream of the M&H Site. The Lake DePue Fish and Wildlife Area and the Spring Lake Heron Colony are situated about 15 miles downstream of the M&H Site. The City of LaSalle obtains its drinking water from a cluster of four wells located 0.75 mile south of the M&H Site, with the nearest municipal well also situated approximately 0.75 mile south of the M& H Site. Nearly 10,000 people live within a 1-mile radius of the M&H Site.

2.1 SITE HISTORY

The M&H Site began operations in 1858 when raw materials such as zinc ore and various grades of coal were transported to smelt zinc. A rolling mill was built on site in 1866 to produce zinc sheets. This process included a furnace that used producer gas as fuel, and any sulfur dioxide generated was recovered and converted into sulfuric acid, which was stored in on-site tanks. The M&H Site also had an ammonium sulfate fertilizer plant that was operational for a few years during the early 1950s. Coal mining occurred at the M&H Site until 1937; two mining shafts (one vertical, one horizontal) remain, although little surface evidence of the mining shafts is apparent at the site today. Zinc smelting ceased in 1961, while sulfuric acid manufacturing halted in 1968. From 1968 until 1978, the facility was taken over by the Zinco Company, and only rolling mill operations were performed. Matthiessen & Hegeler Zinc Company

declared bankruptcy in 1978. This 12-acre tract was purchased by Fred and Cynthia Carus in 1980 and became the LaSalle Rolling Mills.

In 1991, Zinco merged with the LaSalle Rolling Mills, which was the surviving corporation. The LaSalle Rolling Mills worked under contract with the United States Mint to generate metal blanks for pennies and operated until 2000, when bankruptcy was declared. In 2003, EPA conducted an emergency removal at the LaSalle Rolling Mills to address cyanide contamination, the old plating line, and various other chemicals and storage tanks that remained after the rolling mill closure. This removal action is complete.

South of the rolling mills is the Carus Chemical Company and Carus Chemical property. The chemical company has been operational since 1915 and mainly produces potassium permanganate. Wastewater, generated during production of potassium permanganate, is discharged to a treatment pond and eventually into the Little Vermilion River, pursuant to a national pollutant discharge elimination system permit. Solid wastes generated from manufacturing activities are transported off site to a permitted landfill used solely by the Carus Chemical Company.

The M&H Site has been divided into two operable units: OU1 and OU2 (see Table 1). As negotiated by a settlement order signed in September 2006, OU1 includes the Carus Chemical Company and property, the Little Vermilion River adjacent to the entire M&H Site, and a large slag and sinter waste pile, approximately 6 acres in area and 40 to 100 feet high. OU2, approximately 140 acres, is identified as the production area of the former zinc smelting and rolling processes and the immediate property surrounding this area. Specifically, OU2 includes the former rolling mill facility, approximately 150 associated former buildings and structures, a shallow slag and sinter pile that heterogeneously covers the former production area of the M&H Site, several abandoned and closed mine shafts, an undeveloped woodland, and surrounding residential areas. The bulk of the residential area is being investigated by the EPA Field Environmental Decision Support (FIELDS) Team.

TABLE 1
OPERABLE UNITS AT M&H SITE

Operable Unit	Description
OU1	Carus Chemical Company, 6-acre slag pile, and Little Vermilion River
OU2	Former M&H Zinc Company plant excluding OU1. OU2 also includes nearby residential properties that have been contaminated by site-related metals.

Notes:
OU Operable Unit

Sources of metals on the M&H Site are the former smelting process, coal mining operations, and any residuals and byproducts from these processes. The operations included converting the raw zinc ore containing zinc sulfide to zinc oxide and, subsequently, smelting the zinc oxide sinter to produce metallic zinc. The sulfur from the first phase of the process was recovered and converted into sulfuric acid. Much of the equipment associated with sulfuric acid production was either of lead construction or lead lined. A lead burner was used on site for the manufacture and repair of lead components. Other metals were also present in the zinc ore as impurities, including lead and cadmium. A narrow-gauge, on-site industrial railroad was used to move ore about the M&H Site.

Pesticides/herbicides may be found at the M&H Site because in the mid 1900s, the railroads commonly sprayed these types of products for vegetation control; three of those railroads (Illinois Central, LaSalle and Bureau County, and the on-site narrow gauge industrial railroad) were located on the M&H Site. Pesticides also may have been used for control measures during site operations.

Many documented potential organic contaminant sources lead to expectation of potential semivolatile organic compound (SVOC) and volatile organic compound (VOC) contamination at the M&H Site. VOC and SVOC usage have been pervasive at the M&H Site, as detailed in the following list of source candidates for potential soil and groundwater contamination:

- Gasoline-powered locomotives used for moving ore cars on site
- Presence of at least one gasoline underground storage tank
- Producer gas used as a fuel source for some of the kilns area
- Machinery and engine oils used sitewide
- Coal burning on site
- Presence of carbon tetrachloride fire extinguishers

- Presence of research (analytical) laboratories
- Presence of engine/machine shops.

Asbestos and polychlorinated biphenyl (PCB) contamination have also be found in surface soils of OU2. Asbestos was used as a building material (transite walls and roofs), thermal insulation, and fire proofing in many of the 150 M&H buildings. Steam pipes that traversed the M&H Site were wrapped in asbestos type insulation. During at least part of the time that the M&H Zinc Company was in operation, it generated its own electrical power for use in the zinc refining plant and the M&H coal mine. Several transformers are known to have been located on OU2. PCBs were used in electrical transformers manufactured between 1929 and 1977. The removal and disposal of these transformers is not documented; thus, PCB soil contamination may be found in the vicinity of former transformer sites.

Many of the above-listed chemicals of interest possibly were mobilized about the site via wind and routine site operations. Three main railroads were operational during various periods of the M&H Site operations. The railroads traversed the M&H Site north-south, with one railroad (Illinois Central) on the east side and the other (LaSalle and Bureau County) on the west side of the property. Flatbed and open hopper cars crossing the M&H Site, as part of site operations, possibly deposited byproduct and process-derived contamination around the site.

2.2 FORMER SITE INVESTIGATIONS

The M&H Site was listed on the National Priorities List (NPL) on September 29, 2003. Two primary sources located on the property were used to score the site for the NPL. The first source is the 6-acre slag and sinter pile, mostly located on the Carus Chemical Company property of the M&H Site (OU1). This contamination source is addressed by the PRPs, and SulTRAC is providing technical assistance under a separate WA (015-RSBD-B568) and SOW. Therefore, OU1-related investigation activities will not be further discussed in this FSP.

The second source included within OU2 is a shallow waste pile composed of sinter and slag heterogeneously deposited throughout the former smelter property. The contaminants discovered in the second source appear to be the result of former zinc smelter activities and ancillary operations as described previously. Runoff from this shallow sinter and slag waste pile flows into the Little Vermilion River through natural drainage pathways and manmade conduits. For example, in the central portion of OU2, west of the abandoned railroad, a conduit runs from an abandoned pump house north to the Little Vermilion River, and an old abandoned storm sewer line runs east-west across the entire width of OU2.

Three newly discovered (April 2008) outfall pipes in the far northeastern corner, east of the abandoned railroad, empty groundwater over the cliff into the Little Vermilion.

During the November 1991 CERCLA screening site inspection, and the December 1993 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Integrated Assessment sampling, the Illinois Environmental Protection Agency (IEPA) collected several samples from the two sources. Five samples were collected from the sinter slag cover on OU2. The IEPA also observed a release to surface water during the 1993 screening that was subsequently substantiated by chemical analyses of sediment samples in the Little Vermilion River (IEPA 1994).

During the 1993 Assessment, several soil samples collected from nearby residential properties were found to contain elevated levels of metals, primarily cadmium and lead, associated with the M&H Site. The presence of cadmium and lead in the residential soil samples spurred the Agency for Toxic Substances and Disease Registry and the Illinois Department of Public Health (IDPH) to issue a public health statement in September 1999, calling the M&H Site a public health hazard, which was confirmed by laboratory results (IDPH 2000). Trespassers on the M&H Site may also be exposed to the contamination and can easily access the site through large holes in the fence or via the Little Vermilion River.

The preliminary results of the 2007 Phase I OU2 RI show ubiquitous metal contamination across the entire site—namely arsenic, lead, cadmium, copper, mercury, and zinc in soils, debris piles, building materials, surface water, and groundwater. Areas of significant PCB contamination are in debris piles and surface and subsurface soils near Building 100, the rolling mill, and the furnaces. Trichloroethene (TCE) contamination is also found in soils and groundwater in the vicinity of the rolling mill on OU2. Polyaromatic hydrocarbons have been detected ubiquitously throughout OU2. Asbestos has been found in concentrations as high as 6.5 percent in surface soils and fill only. Based on these results, the nature of contamination at the site has been fairly well characterized, and determining the extent and delineation of contamination is a goal of the 2008 Phase II RI.

Identification of the chemicals of interest potentially hazardous to human health and the environment at the M&H Site within OU2 was based on the above-documented investigations and information obtained by SulTRAC. These chemicals of interest are shown in Table 2.

TABLE 2
CHEMICALS OF INTEREST AT OU2 FROM PRIOR SITE INVESTIGATIONS

Chemical of Interest	IEPA Assessment Maximum Surface Concentration[1] (mg/kg)	RI/FS Phase I – Maximum Surface Concentration[2] (mg/kg)	RI/FS Phase I – Maximum Subsurface Concentration[2,3] (mg/kg)
Cadmium	1,320	7,350	770
Copper	3,650	7,020	2,430
Lead	4,310	51,900	62,600
Zinc	71,200	408,000	158,000
Arsenic	36	812	528
Mercury	unknown	154	143
PCB	unknown	150	18
Asbestos	unknown	6.5%	no detections at depth
TCE	unknown	0.01	120

Notes:

[1] Results are based on five samples collected in 1993 (IEPA 1994).
[2] Results are based on 250 samples collected in July and August 2007 (SulTRAC 2008b).
[3] Surface sample depths are 0-2 feet below ground surface.
[4] Subsurface depths range between 8 and 12 feet below ground surface.

mg/kg	Milligrams per kilogram
PCB	Polychlorinated biphenyls
TCE	Trichloroethene
IEPA	Illinois Environmental Protection Agency
RI/FS	Remedial investigation/Feasibility study
OU	Operable Unit

3.0 PROJECT OBJECTIVE

The purpose of this FSP is to describe the approach that will be used to conduct Phase II of the RI/FS at OU2 on the M&H Site. Phase II encompasses field and other activities to finish characterizing contamination detected during the Phase I investigation and to further delineate the extent of contamination. Phase II will also suggest a remedy to eliminate, reduce, or control risks to human health and the environment. After the Phase II investigation is complete, a RI report will be prepared documenting the results of the investigations. The RI report will include a risk assessment that evaluates actual or potential risks to human health and the environment.

A FS will evaluate potential alternatives for site remediation. The RI/FS involves investigation and study of the former rolling mill and associated buildings, the shallow sinter and slag cover that exists over much of the M&H Site, and surrounding residential areas that have been defined as OU2. The residential area is being investigated by the EPA FIELDS Team. The goal is to develop the minimum amount of data necessary to support the selection of an approach for site remediation and then to use this data to attain a well-supported Record of Decision (ROD). All SulTRAC field activities will be conducted in accordance with the EPA-approved, site-specific QAPP (Attachment B) and SulTRAC standard operating procedures (SOP) (see SOP Attachment). Where the FSP differs from the SOPs, the FSP's site-specific procedures will take precedence.

4.0 FIELD SAMPLING ACTIVITY

Field sampling activities discussed in this section pertain to the Phase II RI, which will focus on delineating the extent of contamination and closing any data gaps identified in the Phase I investigation. Figures 2 through 5 show the locations of proposed Phase II sampling activities, and Table 3 lists all samples to be collected and their associated identification and collection specifics. A detailed discussion of sample collection procedures is presented in Section 5.0.

Prior to intrusive field activities, asbestos air samples will be collected inside the rolling mill. As discussed with the EPA and outlined in the OU2 work plan (SulTRAC 2007, 2008a), SulTRAC will conduct Phase II field activities including collecting surface and subsurface soil samples from 60 borings; collecting building material samples from 50 building/structures; installing 17 new monitoring wells and six piezometers; performing soil X-ray fluorescence (XRF) screening and collecting soil samples based on XRF screening; collecting soil, sinter, and slag pile samples for bioassessibility testing; collecting vegetation, earthworm, and soil/sinter/slag samples for bioavailability testing; collecting groundwater samples from existing and the new monitoring wells; performing hydraulic tests to estimate aquifer characteristics; and collecting surface water samples from eight locations. A summary of sample information is presented below in Table 4.

4.1 AIR SAMPLING

Asbestos air sampling will be conducted inside the rolling mill prior to any other field activities. This air monitoring will be conducted in two locations during an active day of Fred Carus's warehousing business, which operates out of this facility. Inside the rolling mill, backer-board is warehoused and distributed with daily pickups of the product via tractor trailer vehicles. Two additional samples will be collected downwind from two locations in the former main industrial plant area at which analytical detections of asbestos in soils occurred. Air samples will be analyzed through a subcontracted laboratory for asbestos. Samples will be analyzed for asbestos using appropriate EPA methods, as identified in Section 6.0 of this FSP.

4.2 SOILS AND DEBRIS PILES

SulTRAC will conduct geological investigations and collect surface and subsurface samples. Surface and subsurface samples will be collected via soil borings (Section 4.2.1) in a biased approach. Approximately

TABLE 3
SAMPLE IDENTIFICATION INFORMATION FOR ALL MATRICES

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB401A-08	Soil boring	Field	SB401	Near Building 100	0-2				x	x											
SB401B-08	Soil boring	Field	SB401	Near Building 100	>12				x	x								x			
SB402A-08	Soil boring	Field	SB402	Near Building 100	0-2				x	x											
SB402B-08	Soil boring	Field	SB402	Near Building 100	>12				x	x								x			
SB403A-08	Soil boring	Field	SB403	Near Building 100	0-2				x	x											
SB403B-08	Soil boring	Field	SB403	Near Building 100	>12				x	x								x			
SB404A-08	Soil boring	Field	SB404	Near Building 100	0-2				x	x											
SB404B-08	Soil boring	Field	SB404	Near Building 100	>12				x	x								x			
SB405A-08	Soil boring	Field	SB405	Near Building 100	0-2				x	x											
SB405B-08	Soil boring	Field	SB405	Near Building 100	>12				x	x								x			
SB406A-08	Soil boring	Field	SB406	Near Building 100	0-2				x	x						x					
SB406B-08	Soil boring	Field	SB406	Near Building 100	>12				x	x						x		x			
SB407A-08	Soil boring	Field	SB407	Near Building 100	0-2				x	x											
SB407B-08	Soil boring	Field	SB407	Near Building 100	>12				x	x								x			
SB408A-08	Soil boring	Field	SB408	Near Building 100	0-2				x	x											
SB408B-08	Soil boring	Field	SB408	Near Building 100	>12				x	x								x			
SB409A-08	Soil boring	Field	SB409	Near Building 100	0-2				x	x											
SB409B-08	Soil boring	Field	SB409	Near Building 100	>12				x	x								x			
SB410A-08	Soil boring	Field	SB410	Near Building 100	0-2				x	x							x				
SB410B-08	Soil boring	Field	SB410	Near Building 100	>12				x	x								x			
SB411A-08	Soil boring	Field	SB411	NW of Rolling Mill	0-2	x				x											
SB411B-08	Soil boring	Field	SB411	NW of Rolling Mill	2-12	x				x											
SB412A-08	Soil boring	Field	SB412	NW of Rolling Mill	0-2	x				x						x					
SB412B-08	Soil boring	Field	SB412	NW of Rolling Mill	2-12	x				x						x					
SB413A-08	Soil boring	Field	SB413	NW of Rolling Mill	0-2	x				x											
SB413B-08	Soil boring	Field	SB413	NW of Rolling Mill	2-12	x				x											
SB414A-08	Soil boring	Field	SB414	NW of Rolling Mill	0-2	x				x											
SB414B-08	Soil boring	Field	SB414	NW of Rolling Mill	2-12	x				x											
SB415A-08	Soil boring	Field	SB415	NW of Rolling Mill	0-2	x				x											
SB415B-08	Soil boring	Field	SB415	NW of Rolling Mill	2-12	x				x											
SB416A-08	Soil boring	Field	SB416	NW of Rolling Mill	0-2	x				x							x				
SB416B-08	Soil boring	Field	SB416	NW of Rolling Mill	2-12	x				x											
SB417A-08	Soil boring	Field	SB417	NW of Rolling Mill	0-2	x				x											
SB417B-08	Soil boring	Field	SB417	NW of Rolling Mill	2-12	x				x											
SB418A-08	Soil boring	Field	SB418	NW of Rolling Mill	0-2	x				x											
SB418B-08	Soil boring	Field	SB418	NW of Rolling Mill	2-12	x				x											
SB419A-08	Soil boring	Field	SB419	NW of Rolling Mill	0-2	x				x											
SB419B-08	Soil boring	Field	SB419	NW of Rolling Mill	2-12	x				x											
SB420A-08	Soil boring	Field	SB420	NW of Rolling Mill	0-2	x				x											
SB420B-08	Soil boring	Field	SB420	NW of Rolling Mill	2-12	x				x											
SB421A-08	Soil boring	Field	SB421	North Area	0-2	x	x	x	x	x				x							
SB421B-08	Soil boring	Field	SB421	North Area	2-12	x	x	x	x	x											
SB422A-08	Soil boring	Field	SB422	North Area	0-2	x	x	x	x	x				x							
SB422B-08	Soil boring	Field	SB422	North Area	2-12	x	x	x	x	x											
SB423A-08	Soil boring	Field	SB423	North Area	0-2	x	x	x	x	x				x		x					
SB423B-08	Soil boring	Field	SB423	North Area	2-12	x	x	x	x	x						x					
SB424A-08	Soil boring	Field	SB424	North Area	0-2	x	x	x	x	x				x							
SB424B-08	Soil boring	Field	SB424	North Area	2-12	x	x	x	x	x											
SB425A-08	Soil boring	Field	SB425	North Area	0-2	x	x	x	x	x				x							
SB425B-08	Soil boring	Field	SB425	North Area	2-12	x	x	x	x	x											
SB426A-08	Soil boring	Field	SB426	North Area	0-2	x	x	x	x	x				x							
SB426B-08	Soil boring	Field	SB426	North Area	2-12	x	x	x	x	x											
SB427A-08	Soil boring	Field	SB427	North Area	0-2	x	x	x	x	x				x			x				
SB427B-08	Soil boring	Field	SB427	North Area	2-12	x	x	x	x	x											
SB428A-08	Soil boring	Field	SB428	North Area	0-2	x	x	x	x	x				x							
SB428B-08	Soil boring	Field	SB428	North Area	2-12	x	x	x	x	x											
SB429A-08	Soil boring	Field	SB429	North Area	0-2	x	x	x	x	x				x							
SB429B-08	Soil boring	Field	SB429	North Area	2-12	x	x	x	x	x											
SB430A-08	Soil boring	Field	SB430	North Area	0-2	x	x	x	x	x				x							
SB430B-08	Soil boring	Field	SB430	North Area	2-12	x	x	x	x	x											
SB431A-08	Soil boring	Field	SB431	Inside Rolling Mill	0-2	x	x	x	x	x				x			x				
SB431B-08	Soil boring	Field	SB421	Inside Rolling Mill	2-12	x	x	x	x	x											
SB432A-08	Soil boring	Field	SB432	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB432B-08	Soil boring	Field	SB432	Inside Rolling Mill	2-12	x	x	x	x	x											
SB433A-08	Soil boring	Field	SB433	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB433B-08	Soil boring	Field	SB433	Inside Rolling Mill	2-12	x	x	x	x	x											
SB434A-08	Soil boring	Field	SB434	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB434B-08	Soil boring	Field	SB434	Inside Rolling Mill	2-12	x	x	x	x	x											

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB435A-08	Soil boring	Field	SB435	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB435B-08	Soil boring	Field	SB435	Inside Rolling Mill	2-12	x	x	x	x	x											
SB436A-08	Soil boring	Field	SB436	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB436B-08	Soil boring	Field	SB436	Inside Rolling Mill	2-12	x	x	x	x	x											
SB437A-08	Soil boring	Field	SB437	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB437B-08	Soil boring	Field	SB437	Inside Rolling Mill	2-12	x	x	x	x	x											
SB438A-08	Soil boring	Field	SB438	Inside Rolling Mill	0-2	x	x	x	x	x				x		x					
SB438B-08	Soil boring	Field	SB438	Inside Rolling Mill	2-12	x	x	x	x	x						x					
SB439A-08	Soil boring	Field	SB439	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB439B-08	Soil boring	Field	SB439	Inside Rolling Mill	2-12	x	x	x	x	x											
SB440A-08	Soil boring	Field	SB440	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB440B-08	Soil boring	Field	SB440	Inside Rolling Mill	2-12	x	x	x	x	x											
SB441A-08	Soil boring	Field	SB441	NE Periphery Area	0-2	x	x	x	x	x				x							
SB441B-08	Soil boring	Field	SB441	NE Periphery Area	2-12	x	x	x	x	x											
SB442A-08	Soil boring	Field	SB442	NE Periphery Area	0-2	x	x	x	x	x				x							
SB442B-08	Soil boring	Field	SB442	NE Periphery Area	2-12	x	x	x	x	x											
SB443A-08	Soil boring	Field	SB443	NE Periphery Area	0-2	x	x	x	x	x				x							
SB443B-08	Soil boring	Field	SB443	NE Periphery Area	2-12	x	x	x	x	x											
SB444A-08	Soil boring	Field	SB444	NE Periphery Area	0-2	x	x	x	x	x				x		x					
SB444B-08	Soil boring	Field	SB444	NE Periphery Area	2-12	x	x	x	x	x						x					
SB445A-08	Soil boring	Field	SB445	NE Periphery Area	0-2	x	x	x	x	x				x							
SB445B-08	Soil boring	Field	SB445	NE Periphery Area	2-12	x	x	x	x	x											
SB446A-08	Soil boring	Field	SB446	NE Periphery Area	0-2	x	x	x	x	x				x							
SB446B-08	Soil boring	Field	SB446	NE Periphery Area	2-12	x	x	x	x	x											
SB447A-08	Soil boring	Field	SB447	NE Periphery Area	0-2	x	x	x	x	x				x							
SB447B-08	Soil boring	Field	SB447	NE Periphery Area	2-12	x	x	x	x	x											
SB448A-08	Soil boring	Field	SB448	NE Periphery Area	0-2	x	x	x	x	x				x							
SB448B-08	Soil boring	Field	SB448	NE Periphery Area	2-12	x	x	x	x	x											
SB449A-08	Soil boring	Field	SB449	NE Periphery Area	0-2	x	x	x	x	x				x							
SB449B-08	Soil boring	Field	SB449	NE Periphery Area	2-12	x	x	x	x	x											
SB450A-08	Soil boring	Field	SB450	NE Periphery Area	0-2	x	x	x	x	x				x			x				
SB450B-08	Soil boring	Field	SB450	NE Periphery Area	2-12	x	x	x	x	x											
SB451A-08	Soil boring	Field	SB451	Main Industrial Area	0-2					x		x	x								
SB451B-08	Soil boring	Field	SB451	Main Industrial Area	2-12					x		x	x								
SB452A-08	Soil boring	Field	SB452	Main Industrial Area	0-2					x		x	x			x					
SB452B-08	Soil boring	Field	SB452	Main Industrial Area	2-12					x		x	x								
SB453A-08	Soil boring	Field	SB453	Main Industrial Area	0-2					x		x	x								
SB453B-08	Soil boring	Field	SB453	Main Industrial Area	2-12					x		x	x								
SB454A-08	Soil boring	Field	SB454	Main Industrial Area	0-2					x		x	x								
SB454B-08	Soil boring	Field	SB454	Main Industrial Area	2-12					x		x	x				x				
SB455A-08	Soil boring	Field	SB455	Main Industrial Area	0-2					x		x	x								
SB455B-08	Soil boring	Field	SB455	Main Industrial Area	2-12					x		x	x								
SB456A-08	Soil boring	Field	SB456	Main Industrial Area	0-2					x			x								
SB456B-08	Soil boring	Field	SB456	Main Industrial Area	2-12					x			x								
SB457A-08	Soil boring	Field	SB457	Main Industrial Area	0-2					x			x			x					
SB457B-08	Soil boring	Field	SB457	Main Industrial Area	2-12					x			x								
SB458A-08	Soil boring	Field	SB458	Main Industrial Area	0-2					x			x								
SB458B-08	Soil boring	Field	SB458	Main Industrial Area	2-12					x			x								
SB459A-08	Soil boring	Field	SB459	Main Industrial Area	0-2					x			x								
SB459B-08	Soil boring	Field	SB459	Main Industrial Area	2-12					x			x								
SB460A-08	Soil boring	Field	SB460	Main Industrial Area	0-2					x			x								
SB460B-08	Soil boring	Field	SB460	Main Industrial Area	2-12					x			x								
SB500A-08	Soil boring	Duplicate	SB406A	Near Building 100	0-2				x	x											
SB500B-08	Soil boring	Duplicate	SB406B	Near Building 100	2-12				x	x											
SB501A-08	Soil boring	Duplicate	SB412A	NW of Rolling Mill	0-2	x				x											
SB501B-08	Soil boring	Duplicate	SB412A	NW of Rolling Mill	2-12	x				x											
SB502A-08	Soil boring	Duplicate	SB423A	North Area	0-2	x	x	x	x	x				x							
SB502B-08	Soil boring	Duplicate	SB423B	North Area	2-12	x	x	x	x	x											
SB503A-08	Soil boring	Duplicate	SB438A	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB503B-08	Soil boring	Duplicate	SB438B	Inside Rolling Mill	2-12	x	x	x	x	x											
SB504A-08	Soil boring	Duplicate	SB444A	NE Periphery Area	0-2	x	x	x	x	x				x							
SB504B-08	Soil boring	Duplicate	SB444B	NE Periphery Area	2-12	x	x	x	x	x											
SB505A-08	Soil boring	Duplicate	SB452A	Main Industrial Area	0-2					x		x	x								
SB505B-08	Soil boring	Duplicate	SB457A	Main Industrial Area	0-2					x		x	x								
SB506-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB507-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB508-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB509-08	Soil boring	Duplicate	TBD	TBD	TBD																

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB510R-08	Soil boring	Rinsate	NA	Week 1	NA	x	x	x	x	x											
SB511R-08	Soil boring	Rinsate	NA	Week 2	NA	x	x	x	x	x											
SB512R-08	Soil boring	Rinsate	NA	Week 3	NA	x	x	x	x	x											
SB513R-08	Soil boring	Rinsate	NA	Week 4	NA	x	x	x	x	x											
SB514R-08	Soil boring	Rinsate	NA	Week 5	NA	x	x	x	x	x											
poly_1_001	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_002	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_003	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_004	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_005	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_006	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_007	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_008	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_009	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_010	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_011	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_012	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_013	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_014	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_015	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_016	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_017	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_018	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_019	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_020	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_021	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_022	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_023	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_024	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_025	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_026	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_027	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_028	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_029	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_030	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_031	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_032	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_033	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_034	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_035	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_036	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_037	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_038	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_039	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_040	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_041	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_042	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_043	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_044	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_045	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_046	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_047	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_048	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_049	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_050	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_051	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_052	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_053	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_054	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_055	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_056	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_057	Surface Soil	Field	poly_1[2]	North Area	0-1																ns

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_1_058	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_059	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_060	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_061	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_062	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_063	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_064	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_065	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_066	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_067	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_068	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_069	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_070	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_071	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_072	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_073	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_074	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_075	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_076	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_077	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_078	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_079	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_080	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_081	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_082	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_083	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_084	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_085	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_086	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_087	Surface Soil	Field	poly_1[2]	North Area	0-1					x							x				x
poly_1_088	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_089	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_090	Surface Soil	Field	poly_1[2]	North Area	0-1					x						x					x
poly_1_091	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_2_001	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_002	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_003	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_004	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_005	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_006	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_007	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_008	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_009	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_010	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_011	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_012	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_013	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_014	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_015	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_016	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_017	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_018	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_019	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_020	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_021	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_022	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_023	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_024	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_025	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_026	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_027	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_2_028	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_029	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_030	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_031	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_032	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_033	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_034	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_035	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_036	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_037	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x						x					x
poly_2_038	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_039	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x							x				x
poly_2_040	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_041	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_042	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_043	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_044	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_045	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_046	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_047	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_048	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_049	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_050	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_051	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_052	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_053	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_054	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_055	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x						x					x
poly_2_056	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_057	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_058	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_059	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_060	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_061	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_062	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_063	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_064	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_065	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_066	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_067	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_068	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_069	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_070	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_071	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_072	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_073	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_074	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_075	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_076	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_077	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_078	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_079	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_080	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_081	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_082	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_083	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_084	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_085	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_086	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_087	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_088	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_6_007	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_008	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_009	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_1_200	Surface Soil	Duplicate	poly_1[2]	Duplicate of poly_1_090	0-1					x											x
poly_2_200	Surface Soil	Duplicate	poly_2[2]	Duplicate of poly_2_037	0-1					x											x
poly_2_201	Surface Soil	Duplicate	poly_2[2]	Duplicate of poly_2_055	0-1					x											x
poly_3_200	Surface Soil	Duplicate	poly_3[2]	Duplicate of poly_3_009	0-1					x											x
poly_4_200	Surface Soil	Duplicate	poly_4[2]	Duplicate of poly_4_008	0-1					x											x
SS001-08	Surface Soil	Field	SS001	Main Industrial Area (Area A)	0-2														x		
SS002-08	Surface Soil	Field	SS002	Main Industrial Area (Area A)	0-2														x		
SS003-08	Surface Soil	Field	SS003	Main Industrial Area (Area A)	0-2														x		
SS004-08	Surface Soil	Field	SS004	North Area (Area B)	0-2														x		
SS005-08	Surface Soil	Field	SS005	North Area (Area B)	0-2														x		
SS006-08	Surface Soil	Field	SS006	NE Periphery Area (Area C)	0-2														x		
SS007-08	Surface Soil	Field	SS007	NE Periphery Area (Area C)	0-2														x		
SS008-08	Surface Soil	Field	SS008	Near Building 100 (Area D)	0-2														x		
SS009-08	Surface Soil	Field	SS009	NW of Rolling Mill (Area E)	0-2														x		
SS010-08	Surface Soil	Field	SS010	Residential Area (Area F)	0-2														x		
SS011-08	Surface Soil	Field	SS011	Area East of River (Area G)	0-2														x		
SS012-08	Surface Soil	Field	SS012	Disturbed Woodland-Grassland	0-2															x	
SS013-08	Surface Soil	Field	SS013	Disturbed Woodland-Grassland	0-2															x	
SS014-08	Surface Soil	Field	SS014	Disturbed Woodland-Grassland	0-2															x	
SS015-08	Surface Soil	Field	SS015	Disturbed Woodland-Grassland	0-2															x	
SS016-08	Surface Soil	Field	SS016	Disturbed Woodland-Grassland	0-2															x	
SS017-08	Surface Soil	Field	SS017	Disturbed Woodland-Grassland	0-2															x	
SS018-08	Surface Soil	Field	SS018	Disturbed Woodland-Grassland	0-2															x	
SS019-08	Surface Soil	Field	SS019	Disturbed Woodland-Grassland	0-2															x	
SS020-08	Surface Soil	Field	SS020	Disturbed Woodland-Grassland	0-2															x	
SS021-08	Surface Soil	Field	SS021	Disturbed Woodland-Grassland	0-2															x	
SS022-08	Surface Soil	Field	SS022	Oak-Hickory Woodland	0-2															x	
SS023-08	Surface Soil	Field	SS023	Oak-Hickory Woodland	0-2															x	
SS024-08	Surface Soil	Field	SS024	Oak-Hickory Woodland	0-2															x	
SS025-08	Surface Soil	Field	SS025	Oak-Hickory Woodland	0-2															x	
SS026-08	Surface Soil	Field	SS026	Oak-Hickory Woodland	0-2															x	
SS027-08	Surface Soil	Field	SS027	Oak-Hickory Woodland	0-2															x	
SS028-08	Surface Soil	Field	SS028	Oak-Hickory Woodland	0-2															x	
SS029-08	Surface Soil	Field	SS029	Oak-Hickory Woodland	0-2															x	
SS030-08	Surface Soil	Field	SS030	Oak-Hickory Woodland	0-2															x	
SS031-08	Surface Soil	Field	SS031	Oak-Hickory Woodland	0-2															x	
SS032-08	Surface Soil	Field	SS032	Savannah	0-2															x	
SS033-08	Surface Soil	Field	SS033	Savannah	0-2															x	
SS034-08	Surface Soil	Field	SS034	Savannah	0-2															x	
SS035-08	Surface Soil	Field	SS035	Savannah	0-2															x	
SS036-08	Surface Soil	Field	SS036	Savannah	0-2															x	
SS037-08	Surface Soil	Field	SS037	Savannah	0-2															x	
SS038-08	Surface Soil	Field	SS038	Savannah	0-2															x	
SS039-08	Surface Soil	Field	SS039	Savannah	0-2															x	
SS040-08	Surface Soil	Field	SS040	Savannah	0-2															x	
SS041-08	Surface Soil	Field	SS041	Savannah	0-2															x	
SS042-08	Surface Soil	Field	SS042	Riverine	0-2															x	
SS043-08	Surface Soil	Field	SS043	Riverine	0-2															x	
SS044-08	Surface Soil	Field	SS044	Riverine	0-2															x	
SS045-08	Surface Soil	Field	SS045	Riverine	0-2															x	
SS046-08	Surface Soil	Field	SS046	Riverine	0-2															x	
SS047-08	Surface Soil	Field	SS047	Riverine	0-2															x	
SS048-08	Surface Soil	Field	SS048	Riverine	0-2															x	
SS049-08	Surface Soil	Field	SS049	Riverine	0-2															x	
SS050-08	Surface Soil	Field	SS050	Riverine	0-2															x	
SS051-08	Surface Soil	Field	SS051	Riverine	0-2															x	
SS052-08	Surface Soil	Field	SS052	Vegetation above ground	0-2															x	
SS053-08	Surface Soil	Field	SS053	Vegetation below ground	0-2															x	
SS054-08	Surface Soil	Field	SS054	Vegetation above ground	0-2															x	
SS055-08	Surface Soil	Field	SS055	Vegetation below ground	0-2															x	
SS056-08	Surface Soil	Field	SS056	Vegetation above ground	0-2															x	
SS057-08	Surface Soil	Field	SS057	Vegetation below ground	0-2															x	
SS058-08	Surface Soil	Field	SS058	Vegetation above ground	0-2															x	
SS059-08	Surface Soil	Field	SS059	Vegetation below ground	0-2															x	

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SS060-08	Surface Soil	Field	SS060	Vegetation above ground	0-2															x	
SS061-08	Surface Soil	Field	SS061	Vegetation below ground	0-2															x	
SS062-08	Surface Soil	Field	SS062	Vegetation above ground	0-2															x	
SS063-08	Surface Soil	Field	SS063	Vegetation below ground	0-2															x	
SS100-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS101-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS102-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS103-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS104-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS105-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS106-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
BM011K-08	Building Material[3,4]	Field	BM011	Ore Storage Building	NA		x	x	x	x				x							
BM012T-08	Building Material[3,4]	Field	BM012	Pottery Building	NA		x	x	x	x				x		x					
BM013Z-08	Building Material[3,4]	Field	BM013	Acid Reservoir 3	NA		x	x	x	x		x		x							
BM014T-08	Building Material[3,4]	Field	BM014	Acid Reservoir 4	NA		x	x	x	x				x							
BM015Z-08	Building Material[3,4]	Field	BM015	System 1&2	NA		x	x	x	x				x		x					
BM016C-08	Building Material[3,4]	Field	BM016	System 1&2	NA		x	x	x	x				x							
BM017C-08	Building Material[3,4]	Field	BM017	System 4	NA		x	x	x	x				x							
BM018K-08	Building Material[3,4]	Field	BM018	Refining Plant	NA		x	x	x	x				x							
BM019C-08	Building Material[3,4]	Field	BM019	Refining Plant	NA		x	x	x	x				x							
BM020K-08	Building Material[3,4]	Field	BM020	Building 100	NA		x	x	x	x				x							
BM021C-08	Building Material[3,4]	Field	BM021	Building 100	NA		x	x	x	x				x			x				
BM022K-08	Building Material[3,4]	Field	BM022	Locomotive Building (PCB Area)	NA		x	x	x	x				x							
BM023K-08	Building Material[3,4]	Field	BM023	System 5	NA		x	x	x	x				x							
BM024C-08	Building Material[3,4]	Field	BM024	System 5	NA		x	x	x	x				x							
BM025W-08	Building Material[3,4]	Field	BM025	Ore Storage	NA	x	x	x	x	x				x							
BM026K-08	Building Material[3,4]	Field	BM026	Ore Storage	NA		x	x	x	x				x							
BM027Z-08	Building Material[3,4]	Field	BM027	System 4	NA		x	x	x	x				x							
BM028C-08	Building Material[3,4]	Field	BM028	System 4/Kiln	NA		x	x	x	x		x		x							
BM029W-08	Building Material[3,4]	Field	BM029	System 3	NA	x	x	x	x	x				x							
BM030C-08	Building Material[3,4]	Field	BM030	Coke Crushing	NA		x	x	x	x				x		x					
BM031K-08	Building Material[3,4]	Field	BM031	Oxide Plant	NA		x	x	x	x				x							
BM032K-08	Building Material[3,4]	Field	BM032	Oxide Plant	NA		x	x	x	x				x							
BM033Z-08	Building Material[3,4]	Field	BM033	Tile Pile West of Acid Tanks	NA		x	x	x	x				x							
BM034T-08	Building Material[3,4]	Field	BM034	Top of Furnaces	NA		x	x	x	x				x							
BM035C-08	Building Material[3,4]	Field	BM035	Concentration Plant	NA		x	x	x	x				x							
BM036Z-08	Building Material[3,4]	Field	BM036	Building 101	NA		x	x	x	x				x							
BM037K-08	Building Material[3,4]	Field	BM037	Building 101	NA		x	x	x	x				x							
BM038C-08	Building Material[3,4]	Field	BM038	Top of Furnaces	NA		x	x	x	x				x							
BM039K-08	Building Material[3,4]	Field	BM039	Furnaces	NA		x	x	x	x				x		x					
BM040K-08	Building Material[3,4]	Field	BM040	South of System 4/Kilns/Outfall	NA		x	x	x	x				x							
BM041C-08	Building Material[3,4]	Field	BM041	Sintering Plant	NA		x	x	x	x				x							
BM042W-08	Building Material[3,4]	Field	BM042	Hoisting Engineer	NA	x	x	x	x	x		x		x							
BM043K-08	Building Material[3,4]	Field	BM043	Building 1943	NA		x	x	x	x				x							
BM044C-08	Building Material[3,4]	Field	BM044	Building 1943	NA		x	x	x	x				x							
BM045K-08	Building Material[3,4]	Field	BM045	Rolling Mill	NA		x	x	x	x				x			x				
BM046W-08	Building Material[3,4]	Field	BM046	Boiler House	NA	x	x	x	x	x				x							
BM047Z-08	Building Material[3,4]	Field	BM047	Rolling Mill	NA		x	x	x	x				x							
BM048C-08	Building Material[3,4]	Field	BM048	Rolling Mill	NA		x	x	x	x				x							
BM049K-08	Building Material[3,4]	Field	BM049	Office Building	NA		x	x	x	x		x		x							
BM050K-08	Building Material[3,4]	Field	BM050	Brick Pile East of Acid Reservoir 8	NA		x	x	x	x				x							
BM051C-08	Building Material[3,4]	Field	BM051	Acid Tank	NA		x	x	x	x				x							
BM052C-08	Building Material[3,4]	Field	BM052	Acid Tank	NA		x	x	x	x				x		x					
BM053K-08	Building Material[3,4]	Field	BM053	Top of Furnaces	NA		x	x	x	x				x							
BM054C-08	Building Material[3,4]	Field	BM054	Acid Tank	NA		x	x	x	x				x							
BM055C-08	Building Material[3,4]	Field	BM055	Acid Reservoir 9	NA		x	x	x	x				x							
BM056Z-08	Building Material[3,4]	Field	BM056	Acid Reservoir 7	NA		x	x	x	x				x							
BM057K-08	Building Material[3,4]	Field	BM057	Acid Reservoir 6	NA		x	x	x	x				x							
BM058W-08	Building Material[3,4]	Field	BM058	Wood Pile North of Acid Tanks	NA	x	x	x	x	x				x			x				
BM059K-08	Building Material[3,4]	Field	BM059	Brick Pile East of Acid Reservoir 8	NA		x	x	x	x		x		x							
BM060Z-08	Building Material[3,4]	Field	BM060	Pump House	NA		x	x	x	x				x							
BM200T-08	Building Material[3,4]	Duplicate	BM012	Pottery Building	NA		x	x	x	x				x							

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Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
BM201Z-08	Building Material[3,4]	Duplicate	BM015	System 1&2	NA		x	x	x	x				x							
BM202C-08	Building Material[3,4]	Duplicate	BM030	System 3 - Coke Crushing	NA		x	x	x	x				x							
BM203K-08	Building Material[3,4]	Duplicate	BM039	Furnaces	NA		x	x	x	x				x							
BM204C-08	Building Material[3,4]	Duplicate	BM052	Acid Tank	NA		x	x	x	x				x							
MW01-mm08	Groundwater[5]	Field	MW-01	Western Border		x	x	x	x	x							x				
MW02S-mm08	Groundwater[5]	Field	MW-02S	Western Border		x	x	x	x	x											
MW02D-mm08	Groundwater[5]	Field	MW-02D	Western Border		x	x	x	x	x											
MW03-mm08	Groundwater[5]	Field	MW-03	Western Border		x	x	x	x	x											
MW04-mm08	Groundwater[5]	Field	MW-04	Northeast of Rolling Mill		x	x	x	x	x											
MW05-mm08	Groundwater[5]	Field	MW-05	West of Furnaces		x	x	x	x	x						x					
MW06-mm08	Groundwater[5]	Field	MW-06	Main Industrial Area		x	x	x	x	x											
MW07-mm08	Groundwater[5]	Field	MW-07	North of Furnaces		x	x	x	x	x											
MW08-mm08	Groundwater[5]	Field	MW-08	South of Furnaces		x	x	x	x	x											
MW09-mm08	Groundwater[5]	Field	MW-09	Main Industrial Area		x	x	x	x	x											
MW10-mm08	Groundwater[5]	Field	MW-10	North of Building 100		x	x	x	x	x											
MW11-mm08	Groundwater[5]	Field	MW-11	Main Industrial Area		x	x	x	x	x											
MW12-mm08	Groundwater[5]	Field	MW-12	South of Acid Reservoirs		x	x	x	x	x											
MW13-mm08	Groundwater[5]	Field	MW-13	North of Pottery Building		x	x	x	x	x											
MW14-mm08	Groundwater[5]	Field	MW-14	North of Acid Reservoirs		x	x	x	x	x							x				
MW15-mm08	Groundwater[5]	Field	MW-15	North Area		x	x	x	x	x						x					
MW16-mm08	Groundwater[5]	Field	MW-16	North Area		x	x	x	x	x											
MW17-mm08	Groundwater[5]	Field	MW-17	Main Industrial Area		x	x	x	x	x						x					
MW18-mm08	Groundwater[5]	Field	MW-18	Next to Little Vermillion River		x	x	x	x	x						x					
MW19-mm08	Groundwater[5]	Field	MW-19	East of Main Industrial Area		x	x	x	x	x											
MW20-mm08	Groundwater[5]	Field	MW-20	East of Main Industrial Area		x	x	x	x	x											
MW21-mm08	Groundwater[5]	Field	MW-21	Next to Pump House		x	x	x	x	x											
MW22-mm08	Groundwater[5]	Field	MW-22	North Area		x	x	x	x	x											
MW23-mm08	Groundwater[5]	Field	MW-23	Main Industrial Area		x	x	x	x	x											
MW24-mm08	Groundwater[5]	Field	MW-24	Main Industrial Area		x	x	x	x	x											
MW25-mm08	Groundwater[5]	Field	MW-25	Main Industrial Area		x	x	x	x	x											
MW26-mm08	Groundwater[5]	Field	MW-26	Western Border		x	x	x	x	x											
MW27-mm08	Groundwater[5]	Field	MW-27	Building 100		x	x	x	x	x											
MW28-mm08	Groundwater[5]	Field	MW-28	East of Building 100		x	x	x	x	x											
MW29-mm08	Groundwater[5]	Field	MW-29	Western Border near Rolling Mill		x	x	x	x	x											
MW30-mm08	Groundwater[5]	Field	MW-30	North of Rolling Mill		x	x	x	x	x											
MW31-mm08	Groundwater[5]	Field	MW-31	North of Rolling Mill		x	x	x	x	x											
MW32-mm08	Groundwater[5]	Field	MW-32	East of Rolling Mill		x	x	x	x	x											
MW33-mm08	Groundwater[5]	Field	MW-33	East of Rolling Mill		x	x	x	x	x											
MW34-mm08	Groundwater[5]	Field	MW-34	South of Rolling Mill		x	x	x	x	x											
MW35-mm08	Groundwater[5]	Field	MW-35	North Area		x	x	x	x	x											
MW50-mm08	Groundwater[5]	Duplicate	MW-15	North Area		x	x	x	x	x											
MW51-mm08	Groundwater[5]	Duplicate	MW-17	Main Industrial Area		x	x	x	x	x											
MW52-mm08	Groundwater[5]	Duplicate	MW-5	West of Furnaces		x	x	x	x	x											
MW53-mm08	Groundwater[5]	Duplicate	MW-18	Next to Little Vermillion River		x	x	x	x	x											
MW54-mm08	Groundwater[5]	Duplicate	MW-22	North Area		x	x	x	x	x											
MW55-mm08	Groundwater[5]	Duplicate	MW-28	East of Building 100		x	x	x	x	x											
MW56-mm08	Groundwater[5]	Duplicate	MW-30	North of Rolling Mill		x	x	x	x	x											
MW57R-mm08	Groundwater[5]	Rinsate	NA	sample group #1		x	x	x	x	x											
MW58R-mm08	Groundwater[5]	Rinsate	NA	sample group #2		x	x	x	x	x											
MW59R-mm08	Groundwater[5]	Rinsate	NA	sample group #3		x	x	x	x	x											
MW60R-mm08	Groundwater[5]	Rinsate	NA	sample group #4		x	x	x	x	x											
SW010-mm08	Surface Water[6]	Field	SW010	Ponded Water South of Building 100	NA	x	x	x	x	x	x				x		x				
SW011-mm08	Surface Water[6]	Field	SW011	Outflow from Main Industrial Area	NA	x	x	x	x	x	x				x						
SW012-mm08	Surface Water[6]	Field	SW012	Mouth-Abandoned Sewer Creek	NA	x	x	x	x	x	x				x						
SW013-mm08	Surface Water[6]	Field	SW013	Teminus-Abandoned Sewer Creek	NA	x	x	x	x	x	x				x						
SW014-mm08	Surface Water[6]	Field	SW014	North Area Standing Water	NA	x	x	x	x	x	x				x	x					
SW015-mm08	Surface Water[6]	Field	SW015	Outfall #1	NA	x	x	x	x	x	x				x						
SW016-mm08	Surface Water[6]	Field	SW016	Outfall #2	NA	x	x	x	x	x	x				x						
SW017-mm08	Surface Water[6]	Field	SW017	Outfall #3	NA	x	x	x	x	x	x				x						
SW018-mm008	Surface Water[6]	Field	SW018	Acid Reservoir	NA	x	x	x	x	x	x				x						
SW050-mm08	Surface Water[6]	Duplicate	SW014	North Area Standing Water	NA	x	x	x	x	x	x				x						

TABLE 3
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
QT001-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT002-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT003-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT004-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT005-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT006-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT007-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT008-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															
QT009-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															
QT010-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT011-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT012-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT013-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT014-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT015-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT016-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT017-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT018-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT019-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT020-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT021-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT022-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT023-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT024-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT025-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT026-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															

Notes:

All sampling locations with sample identification numbers are shown in Figures 2 through 5.

A	Surface sample 0 to 2 ft bgs
B	B: Subsurface sampling depth greater than 2 ft bgs
bgs	Below ground surface
BM	Building material
CN	Cyanide
ft	Feet
GW	Groundwater
Hg	Mercury
ID	Identification
mm	Two-digit month number
MS	Matrix spike
MSD	Matrix spike duplicate
MW	Monitoring well
NA	Not applicable
NE	Northeast
ns	Not screened; sample originally part of sample grid, but in-the-field decisions were made not to screen this location from July 28 to 30, 2008
NW	Northwest
OU	Operable Unit
PCB	Polychlorinated biphenyl
Pest	Pesticides
QT	Trip blank abbreviation for sample ID
R	Rinsate
SB	Soil boring
SPLP	Synthetic precipitation leaching procedure
SS	Soil sample
SVOA	Semivolatile organic analysis
TBD	To be determined
TCLP	Toxicity charateristic leaching procedure
VOA	Volatile organic analysis
X	Sample collected for XRF calibration study; analyzed by Contract Laboratory Program laboratory
XRF	X-ray fluorescence

1	All B depths - a specific 2-ft interval will be determined in the field based on requirements in Section 5.2 of the field sampling plan
2	Poly_1, Poly_2, etc., refer to polygon 1, polygon 2, etc., made up in gridded sampling areas throughout OU2 for the XRF screening and suface soil sampling campaign
3	For building samples, only organic samples (wood) or samples with a stained appearance will also be analyzed for VOCs.
4	Sample ID for building materials must include substrate/material composition in sample identification name as in Table 9 of the field sampling plan (determination to be made in the field).
5	Samples will be collected from all 36 monitoring wells for analysis for VOCs, SVOCs, pesticides, PCBs, metals (including Hg), and cyanide during the initial four quarterly sampling events. After the initial quarterly sampling events, analytical data will be evaluated to determined the chemicals of interest/analyte groups to be sampled for in each well.
6	Surface water samples will be collected in June and October 2008.

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_2_089	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_090	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_091	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_3_001	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_002	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																ns
poly_3_003	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_004	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_005	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_006	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_007	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_008	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_009	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x						x					x
poly_3_010	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_011	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_012	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_013	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_014	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_015	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_4_001	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_002	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_003	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_004	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_005	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_006	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_007	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_008	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x						x					x
poly_4_009	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_010	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_011	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_012	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_013	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_5_001	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x											x
poly_5_002	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_003	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_004	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_005	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_006	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_007	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_008	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x							x				x
poly_5_009	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_010	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_011	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_012	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_013	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_014	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_015	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_016	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_017	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_018	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_019	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x											x
poly_5_020	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_021	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_022	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_023	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_024	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																ns
poly_6_001	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																ns
poly_6_002	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_003	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_004	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_005	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_006	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x

TABLE 4
SUMMARY SAMPLE INFORMATION FOR OU2 M&H SITE

Number of Samplings	Matrix	Depth (feet)
4 locations	Air	Not Applicable
50 locations	Soil[1,2]	12 or Refusal
10 locations	Soil[1,2]	>12
50 locations (XRF)	Soil	Surface
40 locations (Bioavailability)	Soil[3]	Surface
12 locations (Bioavailability)	Soil[4]	Surface
11 locations (Bioassessibility)	Soil	Surface
50 locations	Building materials[5]	Surface
17 locations (Phase II wells)	Groundwater[6,7]	13 to 34
19 locations (Phase I wells)	Groundwater[6,7]	13 to 34
8 locations	Surface Water[8]	Surface

Notes:
[1] See Figure 2 for sampling locations.
[2] Samples will be collected from direct-push technology advanced borings.
[3] Samples will be collected for fauna bioavailability testing.
[4] Samples will be collected for flora bioavailability testing.
[5] See Figure 3 for sampling locations.
[6] See Figure 4 for sampling locations.
[7] Samples will be collected from monitoring wells.
[8] See Figure 5 for sampling locations.

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200 additional surface samples, both soils and debris piles, will be screened via XRF in a gridded sampling approach, with 50 of these samples sent to an independent laboratory for confirmation analyses (Section 4.2.2). For the purpose of this FSP, the term soil boring or surface soil sample can include man-made substrates that have been filled or dumped at the M&H Site. Surface and subsurface soil samples collected at the site from soil borings and as grab samples are expected to include the following man-made substrates/fill: soil, sinter, slag, and debris piles. All field notebook entries and geologic logs must include one of the substrate designations for each sampled horizon unless no man-made substrates are encountered; in that case, the specific lithology type (e.g., shale) shall be identified in both the field notebook and geologic log.

4.2.1 Soil Borings

During the Phase II field investigation, SulTRAC will collect soil and pile samples obtained during the advancement of 60 borings. The primary purpose of the sampling activities is to complete characterization of contamination that began during Phase I and delineate the extent of contamination. The proposed boring locations are at or near the locations of existing or former buildings, railroads, and the Little Vermilion River, as shown in Table 5. These proposed locations (see Figure 2) were selected to supplement Phase I soil sampling results or to provide information where data are limited.

Surface (soil and pile) and subsurface soil samples will be collected from 60 borings at two depth intervals per boring. The 60 soil borings will be located as follows: (1) 10 soil borings will be located in the north area of the OU2 M&H Site; (2) 10 soil borings will be located in the northeast periphery of the OU2 M&H Site (east of the Central Railroad and west of the Little Vermilion River); (3) 10 soil borings will be located in the main industrial area; (4) 10 soil borings will be located around Building 100; (5) 10 soil borings will be located in the exterior northwest corner of the rolling mill; and (6) 10 soil borings will be installed inside the rolling mill (see Figure 2).

Fifty borings will be advanced to a depth of 12 feet below ground surface (bgs) unless refusal is encountered before 12 feet. The remaining 10 soil borings will be advanced to a depth greater than 12 feet bgs depending on PCB concentrations in an area of known PCB contamination at 12 feet bgs. In this known PCB contamination area, near building 100, SulTRAC will be using PCB-specific chemistry field kits to determine PCB concentrations at depths greater than 12 feet. The goal is to continue direct- push technology activities with depth until no PCBs are detected via field kit results. This "clean" deep horizon will then be sampled and sent to the Contract Laboratory Program (CLP) laboratory for verification.

TABLE 5
SOIL BORING LOCATIONS BASED ON OU2 SITE-SPECIFIC INFORMATION

Building Number	Location	Use and Environmental Concern	Number of Soil Borings	Number of Samples
--	North of main Industrial Area	Vegetated area, formerly an area where slag, sinter, and debris were dumped	10	20
--	Northeast Periphery	Natural Area, recently found to contain 3 outflow pipes with flowing water	10	20
--	Main industrial area	Heavy metals found during Phase I. Data gap investigation for Phase II.	10	20
100	Building 100	High PCB concentrations found during Phase I. Three tracks for locomotive repair work.	10	20
	Northwest exterior corner of the rolling mill	TCE found during Phase I investigation.	10	20
1-2-3-4-105-117	Rolling mill (inside)	Rolling zinc, coal fuel, oil-filled transformers, reheated zinc, heavy oil on floor, former plating facility, and current warehouse for cement board storage	10	20

Notes:
TCE Trichloroethene
OU Operating Unit
-- No building structure or multiple building structures are specifically associated with these locations.

All the surface and subsurface samples from the combined 30 soil borings in the north area, northeast periphery area, and inside the rolling mill will be analyzed for target analyte list (TAL) metals (including mercury and cyanide), VOCs, SVOCs, PCBs, and pesticides; only the surface samples will also be analyzed for asbestos. All the surface and subsurface samples from the 10 soil borings located in the main industrial area will be sampled for TAL metals (including mercury and cyanide) and undergo the synthetic precipitation leaching procedure (SPLP); samples from five of those locations will undergo toxicity characteristic leaching procedure (TCLP); samples from the 10 soil boring locations in the exterior northwest corner of the rolling mill will be analyzed for VOCs and TAL metals (including mercury and cyanide). The 10 soil borings located by Building 100 will be analyzed for PCBs and TAL metals (including mercury and cyanide). Table 3 details which 10 samples (5 locations) will be analyzed according to the TCLP analysis and which 20 samples (10 locations) for SPLP analysis. Samples will be analyzed for these analytical groups using appropriate EPA methods, as identified in Section 6.0 of this FSP. QC samples (field duplicate, matrix spike [MS], and matrix spike duplicate [MSD]) will be collected for soils as described in Section 11.0 of this FSP.

4.2.2 XRF Screening and Surface Sampling

During the Phase II field investigation, SulTRAC will coordinate with the EPA's FIELDS team in screening approximately 200 surface soil sampling locations (which may include debris piles) using an Innov-X® XRF analyzer. This screening will be conducted under an unbiased sampling approach using a site sampling grid combined with EPA's rapid assessment tools (RAT). The purpose of the XRF field program is to gain extensive and high spatial resolution metal concentrations for the metals of interest (based on the Phase I results)—arsenic, cadmium, lead, mercury, and zinc. This is particularly important in areas difficult for direct- push technology to access, and for sampling in an unbiased manner during Phase II in order to determine contamination extent. SulTRAC will collect approximately 50 samples from these screened XRF locations for analytical measurement via CLP laboratory. These 50 samples will be analyzed for TAL metals (including mercury and cyanide). These fixed laboratory results will be calibrated against the XRF results at the 50 locations from which the samples had been collected for fixed-laboratory analysis, creating a calibration curve against which all OU2 M&H Site XRF data can be matched. All XRF sample identification information is included in Table 3. Samples will be analyzed for the TAL metals (including mercury and cyanide) analytical groups using the appropriate EPA method, as identified in Section 6.0 of this FSP. QC samples (field duplicate, MS, and MS/MSD) will be collected, as described in Section 11.0 of this FSP.

As a point of note, EPA FIELDS team will also be conducting XRF screening investigations in off-site areas that include the residential areas and areas downwind of the M&H Site, east of the Little Vermilion River. For these areas on the east of the Little Vermilion River, FIELDS will be directed by SulTRAC as to where surface soil should be sampled for CLP analyses (TAL metals (including mercury and cyanide), VOC, SVOCs, PCBs, and pesticides). Selection of these areas will be based on results from M&H Site stack modeling. The stack modeling will be conducted by SulTRAC using air dispersion modeling (AERMOD) considering both dry and wet deposition algorithms based on historical operations information in order to estimate the areas of greatest potential deposition in the downwind area, east of the Little Vermilion River.

4.3 BUILDING MATERIALS

During the Phase II field activities, SulTRAC will collect solid samples from 50 M&H Site buildings to be tested for contamination, mainly with the goal of evaluating future disposal options (see Figure 3). Samples will be analyzed for TAL metals (including mercury and cyanide), SVOCs, PCBs, pesticides, and asbestos. Five additional samples will be analyzed for TCLP. Only organic samples (wood) or samples with a stained appearance will also be analyzed for VOCs. Samples will be analyzed for these analytical groups using appropriate EPA methods, as identified in Section 6.0 of this FSP. QC samples (field duplicate, MS, and MS/MSD) will be collected for building materials, as described in Section 11.0 of this FSP.

4.4 GROUNDWATER

SulTRAC will conduct hydrogeologic investigations involving installation of piezometers, installation and development of new monitoring wells, execution of slug tests, downhole geophysics and resistivity measurements, quarterly sampling of Phase I and Phase II monitoring wells, and surface elevation measurements.

Presently, 19 groundwater monitoring wells are located within OU2. To determine the extent of groundwater contamination, as well as complete groundwater characterization on site, SulTRAC will install an additional 17 monitoring wells and six piezometers (see Figure 4). All new monitoring well and piezometer borings will have downhole geophysics performed prior to the installation of wells. The resulting monitoring well network will be used to identify the nature and extent of contamination within the first water bearing zone at OU2.

Aquifers within this region are represented by sands and gravels within the surficial glacial deposits, and within the underlying permeable sandstone and limestone bedrock formations. The City of LaSalle has a wellfield approximately 0.75 mile south of the M&H Site. This water supply is from the glacial sands and gravels at 60 to 70 feet bgs, well below the current ground surface elevation of the M&H Site. Currently, the ground elevation at the M&H Site ranges between 550 to 650 feet above mean sea level (amsl), and the well field ground elevation is 450 feet amsl. The City of Peru has a wellfield located approximately 2 miles northwest of the M&H Site. This water supply is from the bedrock formation greater than 2,000 feet bgs. Therefore, all newly installed wells at the OU2 M&H Site shall be located in the first water bearing zone. Previous monitoring well installations at the site were set in both Paleozoic and Quaternary sedimentary deposits, some of which likely contained perched aquifers. The groundwater dynamics at the M&H Site are complex because of perched glacial aquifers and the amount of excavation and backfilling that has occurred over the past 150 years. For example, between the November 2007 and March 2008 groundwater sampling, water variability was great. Several wells did not yield enough water to be adequately developed until March 2008, and a previously dry well was able to yield samples. During both sampling events, several developed monitoring wells had such low recharge rates that not enough water was generated to fill all sample jars for the necessary analyses.

The locations of the new monitoring wells and piezometers have been selected based on two OU2 potentiometric groundwater maps that indicate a general groundwater flow to the east and southeast (see Figures 6 and 7). However, a divide occurs on the western portion of the site near Building 100 and the rolling mill, where some groundwater flows to the southwest. SulTRAC has devised a monitoring network that includes upgradient wells (on the M&H Site), wells in likely source areas (main process area), wells downgradient (near the Little Vermilion River and southwest of the site), and piezometers located along the suspected groundwater divide. This network design emphasizes SulTRAC's RI/FS rationale to characterize and to delineate probable migration pathways of potential contaminants. Final monitoring wells and screen depths will be defined in the field. Only estimates can be made concerning these planned depths, since the surficial geology to a depth of approximately 25 feet bgs is very heterogeneous over short distances. All proposed plans are based on the November 2007 and March 2008 OU2 monitoring well logs and cross-sections.

Based on the data evaluation from Phase I (SulTRAC 2008b), 17 monitoring wells and 6 piezometers will be advanced to depths ranging from 15 to 36 feet bgs. Three of the monitoring wells will be advanced to bedrock to obtain site-specific lithologic records of the site and perform downhole geophysics. After

logging and geophysical testing is complete, the three deep borings will be sealed with bentonite to a depth of 15 to 36 feet bgs, and monitoring well installation and development will be completed.

SulTRAC anticipates both the Phase I and Phase II installed groundwater networks will include groundwater sampling for all analytes (unfiltered TAL metals [including mercury and cyanide], VOCs, SVOCs, PCBs, and pesticides) conducted quarterly for one year, to identify possible seasonal changes. After each of the initial four quarterly sampling events, analytical data will be evaluated, and a technical memorandum will be submitted to EPA specifying the chemicals of interest for each well. Based on the chemicals of interest, specific analyte groups will be selected and sampled for the second year of quarterly sampling. Samples will be analyzed for these analytical groups using appropriate EPA methods, as identified in Section 6.0 of this FSP. QC samples (field duplicate, trip blank, MS, and MS/MSD) will be collected for groundwater as described in Section 11.0 of this FSP.

To determine aquifer parameters such as hydraulic conductivity, rising and falling head slug tests will also be performed on at least 12 of the monitoring wells. Wells will be selected to represent horizontal and vertical heterogeneity.

4.5 SURFACE WATER

During the Phase II field investigation, SulTRAC will collect surface water samples on 2 separate days. Two hydrologic investigations will be conducted to sample surface water likely at the beginning of summer and at the end of the summer 2008. SulTRAC is testing seasonal variation, as precipitation variation (during same season) seemingly exerted no influence on surface water during Phase I. Eight samples will be collected during each sampling event (see Figure 5, Table 3). Three drainage pipes discharging water were observed in the northeast corner of the site during a site visit in April 2008. Surface water samples will be collected at these three locations (if water is present), at two locations at the mouth and terminus of the creek emanating from the abandoned sewer line and emptying into the Little Vermilion River, at one location in the north area where standing water is often witnessed, and at two locations of discharge from the main industrial plant area. The purpose of the sampling activities is to identify the nature of contamination in the surface water of OU2.

Samples will be analyzed for total hardness, VOCs, unfiltered and filtered (dissolved) TAL metals (including mercury and cyanide), SVOCs, PCBs, and pesticides. Samples will be analyzed for these analytical groups using appropriate EPA methods as identified in Section 6.0 of this FSP. In addition, QC

samples (field duplicate, trip blank, MS, and MS/MSD) will be collected for surface water as described in Section 11.0 of this FSP.

The first round of Phase II surface water sampling was conducted under conditional approval from the EPA on July 8, 2008. During this first round, samples were not analyzed for total hardness or filtered TAL metals because a comment on this issue was not received from EPA until after the sampling date. These analyses will be included during the second round of Phase II surface water sampling.

4.6 ECOLOGY AND BIOLOGY

Ecological risk assessment will rely on analytical results associated with previously collected Phase I samples as well as Phase II samples. This section focuses on Phase II samples of specific relevance to characterizing potential ecological exposures at the site. These samples are associated with two specific investigations conducted as part of Task 3 (Field Investigation/Data Acquisition) of Phase II. Site-specific vegetation analysis (Section 4.6.1), site-specific soil invertebrate analysis (Section 4.6.2), and soil sampling and laboratory bioavailability tests (Section 4.6.3) are described below.

4.6.1 Vegetation

One of the objectives of the baseline ecological risk assessment (BERA) is to gain an understanding of the potential uptake of site-specific contaminants by native plants in each observed habitat. This information will allow site-specific estimates of potential exposures to the herbivores and omnivores that consume grasses and herbaceous plants at the M&H site. The six vegetation sample pairs (12 samples) will consist of an aboveground sample and an underground sample. This approach will enable the food chain model to better estimate exposures to organisms that feed on the roots or leafy portions of plants. This information, along with the laboratory bioavailability data, will be used in the food chain model of the BERA to provide a range of potential vegetation concentrations that will be used to estimate exposure doses.

4.6.2 Soil Invertebrates

Another objective of the BERA is to provide site-specific information on the potential movement of contaminants within the food chain. The primary goal is to establish site-specific information that may be used to develop site-specific bioaccumulation factors (BAF) to estimate prey tissue concentrations rather than relying on literature-based BAF values. The focus of the soil invertebrate sampling will be earthworms. Earthworms will be collected from soil at five locations within each of the general habitat

areas. A collocated soil sample will also be collected to determine the site-specific BAFs. This information will be used in the food chain model of the BERA to provide a range of potential soil invertebrate concentrations that will be used to estimate exposure doses.

4.6.3 Soil Sampling and Bioavailability Tests

As noted above, one of the BERA objectives is to provide a more site-specific assessment of the bioavailability of contaminants in soils to site receptors, including accumulation of the contaminants in receptor tissues. The goal of the field activities described above is to collect site-specific vegetation and soil invertebrate information. However, there is a concern that it may not be possible to collect a sufficient mass of soil invertebrates or plants needed for tissue analysis from portions of the site. As a contingency, 10 soil samples will be collected from each of the four identified habitats at the M&H site – highly disturbed, disturbed with vegetation, savannah, and oak-hickory woodlands (see Figure 8). These soil samples will be used in 28-day bioavailability tests using earthworms and lettuce seedlings. The earthworm test will be conducted following the ASTM International Standard E 1676-04 (ASTM International 2007). For the plant bioavailability tests, lettuce seedlings will be planted in replicate trays with the number of plants scaled to meet the analytical biomass requirements. The seedlings will be grown for 28 days, and the aboveground portions will be harvested and homogenized to create a single composite sample for each soil sample. The earthworms and vegetation samples will be frozen and shipped to the analytical laboratory to determine tissue concentrations of the contaminants.

4.7 HUMAN HEALTH

The human health risk assessment (HHRA) will rely on analytical results associated with previously collected Phase I samples, as well as Phase II samples. This section focuses on those Phase II samples of specific relevance to characterizing potential human health exposures both on and off site. These samples are associated with four investigations conducted as part of Task 3 (Field Investigation/Data Acquisition) of Phase II. Discussed below is the HHRA-related sampling conducted as part of each of these investigations: air quality investigations (Section 4.7.1); geological investigations (Section 4.7.2); hydrologic and hydrogeologic investigations (Section 4.7.3); and ecological and biological investigations (Section 4.7.4).

4.7.1 Air Quality Investigations

As discussed in Section 4.1, SulTRAC will conduct air quality investigations during Phase II. Asbestos air sampling will be conducted at two locations inside the rolling mill prior to any other field activities

during an active day of Fred Carus's Backerboard business, currently operating out of the rolling mill. The analytical results of these air samples will be used to assess worker safety issues related to Phase II sampling and are not expected to be used directly as part of the HHRA.

On the other hand, SulTRAC will collect asbestos air samples downwind from two locations in the Main OU2 plant area at which analytical detections of asbestos in soil occurred. The analytical results of these air samples will be used in the HHRA to evaluate and quantify the potential air concentrations of asbestos to which human receptors may be exposed in the Main OU2 plant area.

4.7.2 Geological Investigations

As discussed in Section 4.2, SulTRAC will collect 110 surface and 60 subsurface samples from areas of soil and debris piles throughout OU2, as well as off site. These samples will be used to supplement the previously collected Phase I samples. Together, the Phase I and II soil and debris pile samples will be used to characterize chemical concentrations in the following on- and off-site human health exposure areas as shown in Figure 9:

On-Site Exposure Areas

- OU2 main industrial plant area (Area A)
- Wooded area north of main industrial area (Area B)
- Wooded area in the northeast periphery (Area C)
- PCB area near Building 100 (Area D)
- TCE area northwest of rolling mill (Area E)

Off-Site Exposure Areas

- Residential area west of OU2 (Area F)
- Area downwind and east of the Little Vermilion River (Area G)

The location and basis for the majority of the surface and subsurface samples has been discussed (see Section 4.2). However, the samples to be collected in the area downwind and east of the Little Vermilion River warrant additional discussion.

The area downwind (east) and across the Little Vermilion River may have been impacted by deposition associated with historical stack emissions from OU2. Stack modeling will be conducted using AERMOD considering both dry and wet deposition algorithms based on historical operations information in order to estimate the areas of greatest potential deposition in the area downwind (east) and across the Little Vermilion River from OU2. Sample techniques, as discussed in Sections 4.2.1 and 4.2.2, for surface soil

samples will be collected in these areas and analyzed in an off-site laboratory (five samples), as well as via XRF technology.

4.7.3 Hydrologic and Hydrogeologic Investigations

As discussed in Section 4.4 and Section 4.5, quarterly groundwater sampling will occur, and during two separate time periods, eight surface water samples will be collected from throughout OU2 as part of Phase II. These samples will supplement those collected in Phase I and will be used to evaluate the potential for human exposure through groundwater- and surface water-related exposure pathways at OU2.

4.7.4 Ecological and Biological Investigations

The ecological and biological investigations include two types of samples of particular importance to the HHRA. The first is referred to as bioaccessibility samples, which are used to evaluate and quantify the oral bioavailability of metals in soil. Soil samples will be collected from the predominant matrix (e.g., soil, slag, or sinter) or matrices from each of the on- and off-site exposure areas as described in Section 4.7.2. Table 6 presents the number and basis of the soil samples collected in each of the exposure areas.

The soil samples will be analyzed using the relative bioavailability leaching procedure (RBLP) (University of Colorado, Laboratory for Environmental and Geological Studies [LEGS] 2003) (see Section 5.7).

The second type of sample consists of native vegetation collected from areas of known soil contaminant levels. These vegetation samples will be used primarily to support the ecological risk assessment by helping to evaluate and quantify the migration of contaminants from soil (or other solid matrix) up the food chain. Specifically, the analysis of vegetation samples will be used to quantify the uptake of soil-bound metals into vegetation.

As discussed in Section 4.6.2, 40 samples will be collected of native herbaceous vegetation in areas identified, based on Phase I analytical results, as representative of elevated soil contaminant levels. Plants will be extracted from the soil and divided into above ground (including primarily leafs and stems) and below ground (including primarily roots) portions that will be analyzed separately. The savannah habitat evaluated as part of the ecological risk assessment overlaps significantly with the wooded area north of the main plant area adjacent to the off-site residential area. This human health exposure area is considered the most likely to be developed as residential under a future land use scenario. Therefore, 6

vegetation sample pairs, above ground and below ground, will be collected from the savannah habitat to evaluate the potential uptake of soil-bound metals into homegrown produce.

TABLE 6
SUMMARY SAMPLE INFORMATION FOR BIOAVAILABILITY SAMPLES

Human Health Exposure Area	Number and Basis of Soil Samples
OU2 main industrial plant area (Area A)	3 samples – assumes the presence of soil, slag, and sinter
Wooded area north of main industrial area (Area B)	2 samples – assumes the presence of soil and a slag/sinter mix
Wooded area in the northeast periphery (Area C)	2 samples – assumes the presence of soil and a slag/sinter mix
PCB area near Building 100 (Area D)	1 sample – assumes the presence of predominately one type of solid matrix
TCE area northwest of rolling mill (Area E)	1 sample – assumes the presence of predominately one type of solid matrix
Residential area west of OU2 (Area F)	1 sample – assumes the presence of a relatively consistent soil matrix throughout the neighborhood
Area downwind and east of the Little Vermilion River (Area G)	1 sample – assumes the presence of a relatively consistent soil matrix throughout the downwind area
Total	11 samples

Notes:
TCE Trichloroethene
OU Operating Unit
PCB Polychlorinated biphenyl

5.0 FIELD SAMPLING PROCEDURES

This section describes the procedures to be used to collect the types of samples described in Section 4.0. Specifically, this section details the procedures and methods that will be used to collect air, soils, building materials, groundwater, surface water, ecological and biological, and human health samples.

5.1 AIR SAMPLING

Prior to Phase II field activities, asbestos air sampling will be conducted using high-volume air samplers (SOP 064, 073). This air monitoring will be conducted in two locations inside the rolling mill and in two locations downwind from former main industrial plant area at which analytical detections of asbestos in the soils occurred. The air samplers will use a 25-millimeter (mm)-diameter, three-piece cassette loaded with a mixed cellulose ester (MCE) filter of pore size 0.45 micrometers (μm); the filter should be backed by a 5-μm pore size MCE filter. Air sampler pump rates should be calibrated at the start of the day. Sampling will be conducted for eight hours on a day with no precipitation. Calibration rates and start and end times should be noted in the field notebook.

5.2 SOIL AND DEBRIS PILES

Soil samples will be collected at the OU2 M&H Site to complete characterization of contamination that began during Phase I and to delineate the extent of said contamination. The following subsections describe the sampling collection procedures and methods that will be used during the Phase II field investigation.

5.2.1 Soil Borings

During the Phase II field investigation, surface and subsurface soil samples will be collected from 60 borings at two-depth intervals per boring as follows. Prior to sampling, public utility clearance to the site will be accomplished. A private utility locator will be used to scan the area around and inside the rolling mill. SulTRAC also anticipates hiring a company to survey the abandoned sewer line via camera/video communication along the entire length prior to any intrusive activities in this area.

All soil borings will be advanced using hydraulically driven direct-push technology to collect soil samples at specific depths bgs. The 40 sample boring locations located around building 100, the exterior northwestern corner of the rolling mill, inside the rolling mill, and around the main industrial area can be sampled by a direct- push technology rig. However, the 10 samples located in the north area of the M&H Site must be accessed with a direct- push technology devuce mounted to an all-terrain vehicle (ATV), and

the remaining 10 soil boring locations in the northeastern periphery will have to be accessed via a hand auger or hand powered direct- push technology -type machine. All sample collections and geologic logging will be conducted by personnel wearing non-powdered nitrile gloves.

All soil borings will be logged using the SulTRAC geologic logging forms. For each soil boring location, the following information must be included on the logging form: site name, project name, boring number, drilling method, boring diameter, depth to water, date started, date completed, geologist's initials, drilling subcontractor name, and location sketch (with adequate information to find boring location if warranted) with a north directional arrow. During the actual direct- push technology activities, the time of each collected interval, depth (in 2-foot divisions), drive interval, recovered interval, and organic vapor measurements will be recorded on the geologic logging form. The lithologic description will also be recorded for every interval and must include color, texture, and lithology. If slag, sinter, soil, or debris piles are encountered, this information will be clearly specified on the log sheet and the respective field notebook. If more than one sheet is used, the information must be repeated, and the sheets consecutively numbered. All soil boring sample identifications (according to Table 3) will be entered in the appropriate depth interval on the log. All soil boring intervals will be photographed with a tape measure for scale, and the sample identification and depth written on a whiteboard. Photographs will be archived.

Boring samples will be continuously collected from the vadose zone with a disposable acetate sample tube. A 2-foot sample will be collected from the following intervals: 0 to 2 feet bgs and 2 to 12 feet bgs; or to refusal. These samples will be collected using a large bore, dual tube, stainless steel sampler. The sampler will be pushed to the desired depth (from the surface to the total depth of the boring), and then retrieved to collect soil samples. Upon retrieval from the sampler, soil samples will be divided based on sampling intervals for description, screening, and packaging (see SOP 005, SOP054).

Two samples will be collected from each soil boring location. The surface sample, 0 to 2 feet bgs, will be labeled with "A" after the soil location name as indicated in Table 3 and Table 9. The subsurface sample (from the 2- to 12-foot range) will be labeled with "B" after the soil boring location name, and for analytical submission, will be selected based on the interval that appears to have the highest contamination from both field observations and photoionization detector (PID) screening. Soil samples will be screened for visual coloration changes and for organic vapors using a PID. In the event that field screening and observations do not identify an interval to be sampled between 2 to12 feet bgs, SulTRAC will by default collect samples for analysis from the shallowest of the following: above the water table, above bedrock, or the 8- to10-foot interval.

PID soil sample screening will be conducted in the field in the following manner:

- After soil boring retrieval, each sample will be split in half. One half will be placed in a glass container with a teflon-lined lid and placed in an iced cooler for laboratory analysis submittal. The jar will be filled so that no headspace is visible in the container.
- The second half will be placed in a resealable plastic bag, sealed, and vigorously shaken.
- Following a period of approximately 5 minutes for accumulation of organic vapors, the resealable plastic bag will be shaken again. The PID probe will be inserted through a small opening in the plastic bag. After screening, the portion of the sample subjected to headspace screening will be placed with the borehole cuttings for disposal.

Samples for VOC analyses will be collected first, placed directly into the appropriate sample container leaving no headspace, followed by sample collections for TAL metals (including mercury and cyanide), SVOCs, PCBs, pesticides, and asbestos (see SOP 005, SOP 054, SOP 006, SOP S014).

In the vicinity of building 100, known PCB contamination extends down to 12 feet bgs. The subsurface soil boring sampling in this area will be advanced to a depth greater than 12 feet. SulTRAC plans on performing in-field PCB detection tests at these deep depths with the aim of finding a "clean" soil horizon. Once the "clean" horizon is found, this will be the maximum direct- push technology subsurface depth that SulTRAC will sample (SOP PCB). All PCB field kit results will be documented in the field notebook.

All drilling and sampling equipment in contact with soils will be cleaned with a pressurized, hot water sprayer. During sampling operations, sampling equipment will be cleaned using a non phosphate detergent (e.g., Alconox) wash and a potable water rinse. Drilling and sampling equipment will be allowed to air dry following decontamination.

5.2.2 XRF Screening and Surface Sampling

During the Phase II field investigation, SulTRAC and EPA FIELDS team will devise polygon grids for the M&H Site of the areas that need to be screened based on Phase I results. Two teams of two with RAT will begin screening surface soil and debris pile samples via XRF according to EPA Method 6200 (see SOP XRF). The main metal chemicals of interest are arsenic, cadmium, lead, copper, mercury, and zinc. In order to create a usable relationship between the analytical results and the XRF field screening data, approximately 50 samples will be collected from 200 locations across the site with varying contaminant concentrations in order to create a calibration curve. EPA FIELDS will create the calibration curve based on their sampling and analysis plan (EPA 2006a). Sample IDs are specified in Table 3 and Table 9.

Use of the XRF method is restricted to personnel trained and knowledgeable in the operation of a XRF instrument. The XRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. For measurement, the soil sample is placed in a plastic bag, positioned in front of the probe window, and measured. The probe window is placed in direct contact with the plastic bag, mainly to preserve the XRF window quality (see SOP XRF).

After the soil sample has been screened using the XRF, the 50 soil samples will be submitted to a CLP laboratory for TAL metals analysis, including mercury and cyanide.

5.3 BUILDING MATERIALS

Building material samples will be collected at the M&H Site. Fifty building material (brick, concrete, wood, and other) samples will be collected primarily for future waste disposal criteria. All 50 building samples will be analyzed for TAL metals (including mercury and cyanide), SVOCs, PCBs, pesticides, and asbestos. Of these 50 samples, 5 will undergo VOC analysis and five will undergo TCLP analysis.

All sample material types (wood, stone, concrete, brick, etc.) will be described in the field notebook. All samples will be photographed; all locations will be noted in the field notebook, later to be surveyed by professional surveyors.

Collection of grab samples will be accomplished using stainless steel or disposable spades, shovels, and/or scoops where possible. Other tools, such as hammers, chisels, saws, or other cutting tools may be used depending on the building material to be sampled. All tools will be either sterile/disposable or have been precleaned with a non-phosphate cleanser and deionized water rinse. All wood or stained samples will be sampled for VOCs. For samples collected specifically for either VOC analyses, oversized material should be obtained. The sample will be wrapped entirely in aluminum foil and placed in a ziplock-type plastic bag. SulTRAC must instruct CLP laboratory (VOCs) to collect a sub-sample for analysis.

The 50 samples of building material that will be analyzed for TAL metals (including mercury and cyanide), SVOCs, PCBs, and pesticides must be homogenized and ground into pieces as small as possible. Because of the nature of the building material (stone, brick, and concrete), this grinding will be difficult to achieve in the field and the CLP laboratory will likely need to grind the samples into a homogenous powder under a modified analysis. Asbestos samples will be collected whole and sent to a subcontracted laboratory. Specific procedures to collect these types of grab samples are summarized below.

- Use precleaned scoop or trowel to remove vegetation, and then collect the desired volume of solid from the sampling area.
- Label the sample container (ziplock-type bags), and record appropriate data on soil sample data sheets (depth, location, color, and other observations).
- Transfer the discrete grab sample into labeled, ziplock-type bags. All samples, except those to be analyzed for TAL metals (including mercury and cyanide) and asbestos, will be wrapped in clean aluminum foil.
- If the sample is too large to fit into the plastic bag, attempt to manually grind and homogenize the sample or use a precleaned hammer to break up solids.
- Close each sample container as it is filled, clean the exterior of the plastic bag to remove any residue, and then place all samples in a secondary (double-bagged) sample container (ziplock-type bag).
- Place the sample container into an iced cooler maintained at a temperature of 4 degrees Celsius (°C) or lower.

Samples for VOC analyses will be collected first, followed by sample collections for TAL metals (including mercury and cyanide), SVOCs, PCBs, pesticides, and asbestos (SOP 007, SOP S014). Anticipated sub-sampling must be indicated on the chain of custody (COC) for the CLP Laboratory. This analysis will be a modified analysis by CLP.

5.4 GROUNDWATER

Groundwater samples will be collected at the M&H Site. The following subsections describe the procedures to augment the current monitoring well network, as well as sampling collection procedures and methods that will be used during Phase II field investigation.

Presently, 19 groundwater monitoring wells are located within OU2. To determine the extent of groundwater contamination, as well as complete groundwater characterization on site, SulTRAC will install an additional 17 monitoring wells and six piezometers (see Figure 4) to depths ranging from 15 to 36 feet bgs. All new monitoring well and piezometer borings will have downhole geophysics performed. Prior to installations at locations MW25, MW35, and P1 (see Figure 4), resistivity measurements will occur. Alterations to actual proposed monitoring well depths and locations may be determined by the field

team leader (FTL) after actual water table depths are determined. All proposed sample monitoring wells have been mapped (see Figure 4).

5.4.1 Monitoring Well Installation

Monitoring well installation will be performed using either hollow stem or rotosonic augering. All borings will be logged using the SulTRAC geologic logging forms. For each monitoring well boring location, the following information must be included on the logging form: site name, project name, boring number, drilling method, boring diameter, well casing diameter, depth to water, date started, date completed, geologist's initials, drilling subcontractor name, and location sketch (with adequate information to find boring location if warranted) with a north directional arrow. During the actual drilling activities, the time of each collected interval, depth (in 2-foot intervals), drive interval, recovered interval, blow count, and organic vapor measurements will be recorded on the geologic logging form. The lithologic description will also be taken for every interval and must include color, texture, and lithology. If slag, sinter, soil, or debris piles are encountered, this information will be clearly specified on the log sheet and the respective field notebook. If more than one sheet is used, the information must be repeated and the sheets consecutively numbered. All monitoring well boring intervals will be photographed with a tape measure for scale, and depth written on a whiteboard. Photographs will be archived.

For each monitoring well installation, the SulTRAC monitoring well completion record form will be filled out by the SulTRAC geologist on site. This record includes time and date of installation, drilling company, drilling method, and specifics regarding the bentonite seal, filter pack, monitoring well, casing, well screen, annual seal, and borehole backfill (if appropriate).

Samples will be collected continuously for lithologic logging purposes, with downhole geophysics conducted at each monitoring well and piezometer location. The constructed well will be a 2-inch-diameter polyvinyl chloride (PVC) with a 10-foot-long screen and with 0.010-inch slot size. The PVC will allow suitable recharge in low permeability formations and over seasonal water table fluctuations, and will not interfere with the metals analytical sampling.

At three locations (P1, MW25, MW35), three deep borings will be bored out beyond the water table until the massive fossiliferous limestone bedrock is encountered. These deep borings are for the purpose of gaining greater lithologic understanding and resolution at depths beyond 30 ft bgs, across the site, to construct accurate cross-sections and develop a better conceptual site model. These three deep borings will also have resistivity measurements performed within. After logging and geophysical testing is complete, the three deep borings will be sealed with bentonite to a well depth of approximately16 to 46 feet bgs, as

appropriate, and converted to monitoring wells. Monitoring well installation and development will be completed as described below.

Based on quarterly groundwater sampling, current water levels in the monitoring wells range from 4 to 30 feet bgs. In OU2, well screens will be constructed 5 feet above first-encountered groundwater. Clean filter pack will consist of coarsely graded sand that will be installed by pouring from the surface through a tremie pipe from the interval 1 foot below to 1 foot above the well screen. The sand will be poured slowly, and the level of the sand will be periodically tested with a weighted steel tape to prevent bridging. A 2-foot-thick seal of bentonite pellets will be installed at the top of the filter pack, and the annular seal space from the top of the bentonite seal to the surface will be filled with bentonite chips. Following the bentonite seal, a bentonite grout will be emplaced with a tremie pipe from the top of the bentonite seal to the surface. Surface completion will consist of a concrete pad with a 4-inch steel outer protective casing that rises approximately 2 feet above grade. Wells will be completed with expandable locking caps and three bumper posts surrounding each well location (SOP 020). After well installation, all new wells will be surveyed for ground surface elevation, top of casing (TOC) elevation, and horizontal location by a licensed surveyor.

5.4.2 Well Development

Well development will be performed at the newly installed wells. Groundwater well development will consist of surging and bailing to remove fine sediments, followed by pumping to remove approximately five well volumes of water from each well. Wells will be considered developed when the following parameters are stabilized within 10 percent over three consecutive well volumes: turbidity, dissolved oxygen, pH, temperature, and conductivity. Development will be performed using a well development rig, capable of bailing, purging, and pumping groundwater (SOP 021). All purge water will be containerized, labeled, and staged with other investigation-derived waste (IDW) at the M&H Site.

5.4.3 Groundwater Monitoring

The groundwater sampling program involves sampling all monitoring wells for all analytes for the initial four quarters of sampling for either Phase I or Phase II installed monitoring wells. After the initial four quarterly sampling events, analytical data will be evaluated, and a technical memorandum will be submitted to EPA outlining the chemicals of interest for each well. Based on the chemicals of interest, specific analyte groups will be selected and agreed upon by SulTRAC and EPA. These new analyte groups will be sampled for the second year of quarterly sampling. After the second year of sampling, the future sampling frequency will be re-evaluated. Therefore, SulTRAC anticipates writing two groundwater sampling/analytical results technical memorandums for the groundwater monitoring well

network. This applies for both Phase I and Phase II installations. All sampling criteria and sample identifications are listed in Table 3 and Table 9.

Based on the WA032 duration, the Phase II field investigation will include a total of seven quarters of groundwater sampling of which six of the seven events will include both Phase I and Phase II installed wells and piezometers. The first event, scheduled for June 2008, will entail sampling the 19 existing wells. For the remaining sampling events, 36 wells will be sampled, including measuring surface water elevation in these monitoring wells and six piezometers, as well as performing CLP sample paperwork.

Low flow techniques will be used to obtain groundwater samples from all monitoring wells. Low flow, also known as micropurging, provides a method of minimizing increased colloid mobilization by removing water from the well at the screened interval at a rate that preserves or minimally disrupts steady state flow conditions in the aquifer. The well water will be considered stabilized after three successive measurements of field parameters at 3-minute intervals fall within the following ranges: ±0.1 for pH, ±0.5°C for temperature, ±3% for conductivity, ±10 millivolts for oxidation-reduction potential, and ±10% for turbidity and dissolved oxygen (SOP 010, SOP014, SOP 015).

The following information will be recorded in the field logbook at each groundwater sampling location: date and time, barometric conditions, temperature and general weather conditions, depth to water measured from the surveyed top of the well casing, depth to bottom of well measured from the surveyed top of the well casing (SOP 010, SOP014, SOP 015).

Sampling will be performed using the same equipment used for purging. All field measurements will be documented in the field logbook. At each location, groundwater samples will be collected for VOCs analysis first, followed by sample collection for TAL metals (including mercury and cyanide), SVOCs, PCBs, and pesticides. Samples will be directly poured into appropriate (glass or high-density polyethylene) containers (see Table 8). Samples will be immediately placed in an iced cooler, and maintained at a temperature of 4 ± 2°C without freezing until they are delivered to the laboratory under standard COC protocol.

5.4.4 Slug Testing

To determine aquifer parameters such as hydraulic conductivity, slug tests will also be performed. Of the 36 total wells, SulTRAC will slug test at least one-third of the monitoring wells, to ensure results reflecting representative M&H Site conductivity data. The 12 monitoring wells or piezometers selected for the slug tests will be spatially representative of the aquifer both laterally and vertically, and will be chosen by the project manager after analytical results are obtained from the first round of sampling 36 monitoring wells.

The slug testing will likely occur during the September 2008 round of groundwater sampling (see Slug Test SOP Attachment).

5.5 SURFACE WATER

Two hydrologic investigations, eight samples each, will be conducted to sample surface water in June and September 2008, as detailed in Section 4.5. Sample locations are shown in Figure 5. These locations include three drainage pipes in the northeast corner of the site (if water is present), the mouth and terminus of the creek emanating from the abandoned sewer line, a seasonal "pond" in the north area where standing water is often witnessed, and two locations at which discharge occurs from the main industrial plant area.

Surface water will be collected directly into sample containers. The opening of the container will face down, and the container will be slowly and partially submerged beneath the surface of the water and allowed to fill. If the sample container is too large to submerge for collection, an intermediate container, such as the container lid, will be used for sample collection (SOP 009). Also, an intermediate glass container may be used for the VOC collection to prevent the loss of the preservative while submerging. A field peristaltic pump similar to a Geopump® will also be required to sample for dissolved metals using an in-line filter. This type of peristaltic pump, with a low vacuum setting, can also be used to sample for other parameters as long as sediment disturbance is minimized.

When samples are collected for VOC analyses, the container will be checked for air bubbles. If air bubbles are present, additional surface water will be collected in the bottle cap and slowly poured into the vial to remove air bubbles. The container will be recapped and checked again for air bubbles. This procedure will be repeated until no air bubbles are present (SOP 009).

All surface water locations will be photographed with a tape measure for scale and depth, and sample ID will be written on a whiteboard. Photographs will be archived. The sample collection method, date, time, ambient temperature, and visual water characteristics will be documented in the field notebook.

Samples for VOC analyses will be collected first and placed directly into the appropriate sample container leaving no headspace. Samples will be collected next for filtered and unfiltered TAL metals (including mercury and cyanide), SVOCs, PCBs, pesticides, and total hardness (SOP 009). All samples will be immediately placed in an iced cooler and maintained at a temperature of 4 ± 2 °C, without freezing, until delivered to the laboratory under standard COC protocol.

As mentioned in Section 4.5, first round Phase II surface water samples were not collected for total hardness or filtered TAL metals analyses. EPA recommended these analyses after conditional approval of this FSP on June 30, 2008, and after the sampling event was conducted on July 8, 2008. These analyses will be included in the second round of Phase II surface water sampling.

5.6 ECOLOGY AND BIOLOGY

This section focuses on the field sampling procedures associated with Phase II sampling relevant to characterizing potential ecological exposures in the various habitats at the M&H site. As discussed in Section 4.6, these samples are associated with the four habitats identified at the M&H site – highly disturbed, disturbed with vegetation, savannah, and oak-hickory woodlands. Procedures for vegetation sampling (Section 5.6.1), soil invertebrate sampling, (Section 5.6.2) and soil sampling collecting for the bioavailability tests (Section 5.6.3) are discussed below.

5.6.1 Vegetation

The objective of the vegetation sampling is to collect samples of both aboveground leaves and stems and belowground roots from each habitat. As noted in Section 4.6.1, bioavailability information for the aboveground leaves and belowground roots will allow better estimates of potential exposures of the herbivores and omnivores in the food chain model. Three vegetation samples will be collected from each habitat. Because of the lack of vegetation in the highly disturbed area, no samples will be obtained from this habitat. The vegetation samples in the remaining three habitats will be collected as summarized below.

1. Phase I data will be reviewed to locate areas with soil contamination data that indicate contamination both above and below the screening-level ecological risk assessment (SLERA) screening values. Locations will be chosen where results indicate between 0.5 and 3 times the soil screening values in order to obtain vegetation from a range of locations within a habitat area. The goal is to obtain vegetation from areas with known soil contaminant concentrations. If it is not possible to collect sufficient samples from these locations, samples will be collected from locations that will be sampled as part of the Phase II sampling activities.

2. Samples of native species will be collected if available. If they are not available, invasive species will be sampled. This decision will be made by the field biologist at the time of the sampling.

3. The aboveground portions of grasses will be cut from the plant using stainless-steel shears. For herbs, the leaves will be removed from the plant using stainless-steel shears. The aboveground vegetation sample will be placed in plastic ziplock-type bags. The belowground portions of the plants will be dug from the ground using a stainless-steel garden trowel. The roots will be first shaken and then brushed to remove as much loose soil as possible. Next, the roots will be rinsed first with tap water and finally with de-ionized water to remove any remaining soil. The roots

will then be blotted dry with a paper towel to remove as much moisture as possible. The belowground portion of the plant will then be placed in ziplock-type bags.

4. As part of the collection process, the sampler will note the presence or absence of soil invertebrates and, if present, their relative abundance in the soil.

5. Each sample will contain at a minimum 50 grams wet weight of aboveground plant matter and 50 grams wet weight of belowground plant matter. Sava (1994) estimated an adequate sample size of 30 to 40 small leaves, 20 to 25 medium leaves, and 15 to 20 large leaves. Addition of stems will reduce the number of leaves required.

6. The samples will be preserved in a cooler with dry ice and submitted to the analytical laboratory.

5.6.2 Soil Invertebrate Sampling

The objective the soil invertebrate sampling is to collect earthworm tissue samples from each habitat. As noted in Section 4.6.2, the earthworm tissue sample results will be used to estimate the BAFs for soils and provide information on the contaminant body burden in each habitat. This information will be used to allow better estimates of potential exposures of the carnivores and omnivores in the food chain model. Because of the lack of vegetation in the highly disturbed area, no soil invertebrates are expected to be present in this area and no samples will be obtained from this habitat. The soil invertebrate samples in the remaining three habitats will be collected as summarized below.

1. Phase I data will be reviewed to locate areas with soil contamination data that indicate contamination both above and below the SLERA screening values. Locations will be chosen where results indicate between 0.5 and 3 times the soil screening values in order to obtain soil invertebrates from a range of locations within a habitat area. The goal is to obtain soil invertebrates from areas with known soil contaminant concentrations. If it is not possible to collect sufficient samples from these locations, samples will be collected from locations that will be sampled as part of the Phase II sampling activities.

2. Soils will be turned over with a shovel, and earthworms will be collected in a stainless-steel pan or bowl. At least 75 worms are required from each location.

3. Once an adequate number of earthworms have been collected, they will be transported to the work trailer. The worms will be rinsed with tap water to remove soil.

4. The rinsed worms will be placed in a plastic container lined with wet filter paper and then covered. The worms will be allowed to purge their guts for 24 hours (ASTM International 2007).

5. The purged worms will be rinsed again with de-ionized water and placed in a ziplock-type bag. One bag will be used for all worms from the same sampling location.

6. The samples will be preserved in a cooler with dry ice and submitted to the analytical laboratory.

5.6.3 Soil Sampling for Bioavailability Tests

Sub-samples of the soil samples for the bioavailability tests described in Section 4.6.3 will be analyzed to determine contaminant concentrations. The testing will occur concurrently with the bioavailability tests. Soil samples from each habitat will be collected as summarized below.

1. Phase I data will be reviewed to identify areas of known contamination in each habitat. Locations will be chosen where results indicate between 0.5 and 3 times the soil screening values. Also, sample collection will be attempted in areas containing the most common soil type in each habitat.

2. All soil samples will be collected from 0 to 12 inches bgs using a stainless-steel scoop. All vegetation will be removed from the soil surface prior to sample collection.

3. Five equal samples of approximately 3 liters of soil will be collected from each habitat area. The soil from each habitat area will be placed in a 5-gallon plastic bucket and homogenized using a large stainless-steel mixing spoon to break up large clumps. A lid will then be placed on the bucket, and the bucket will be rolled back and forth for 3 minutes or until the soil is thoroughly mixed. The minimum final volume of soil should be approximately 3 gallons. Once homogenized, the soil will be transferred to 1-gallon polyethylene containers, stored at 4 °C, and shipped to the biological testing laboratory.

Before the soils are used for the bioavailability testing, they will be screened by the laboratory conducting the bioavailability tests to determine if contaminant concentrations (1) are sufficiently high to cause lethality to the earthworms and (2) will not allow sufficient germination rates of the lettuce seedlings to allow a successful 28-day test. If greater than 20 percent earthworm lethality is observed after 48 hours, the soils will be diluted with standard laboratory soil. Also, if sufficient lettuce seedling lethality (greater than 10 percent) is observed during the preliminary test, the soils will be diluted with standard laboratory soil to ensure adequate growth to allow completion of the bioavailability test.

5.7 HUMAN HEALTH

This section focuses on the field sampling procedures associated with the Phase II samples relevant to characterizing potential human health exposures both on site and off site. As identified in Section 4.7, these samples are associated with four investigations including air quality, geological, hydrologic and hydrogeologic, and ecological and biological investigations. The field sampling procedures associated with the air quality (Section 5.1), geological (including XRF samples) (Section 5.2), and hydrogeologic and hydrologic (Sections 5.4 and 5.5) investigations have already been presented. With regards to the ecological and biological investigations, Section 5.6.2 has discussed the field sampling procedures associated with collecting native herbaceous vegetation in order to evaluate and quantify uptake of soil-

bound metals into vegetation. Therefore, the remainder of this section will focus exclusively on the field sampling procedures associated with collection of soil samples for analysis by the RBLP.

As discussed in Section 4.7, 11 soil samples will be collected from the predominant matrix (e.g., soil, slag, or sinter) or matrices in the seven on- and off-site exposure areas (see Figure 9) to be evaluated in the HHRA. The sample collection and handling procedures for these soil samples are the same as described in Section 5.2 for soil samples analyzed for metals (SOP 005). The collected samples will be shipped to the LEGS in the Department of Geological Sciences at the University of Colorado, Boulder, where they will be analyzed using the RBLP (also referred to as the SOP In-Vitro) (LEGS 2003). The SOP for the In-Vitro method is presented in the SOP section at the end of this report.

6.0 LABORATORY ANALYTICAL METHODS

Table 7 presents the laboratory methods that will be used to analyze the samples collected by SulTRAC. Field investigation samples will be analyzed by the CLP laboratory and Central Regional Laboratory (CRL).

TABLE 7
ANALYTICAL METHODS SUMMARY

Parameter	Analytical Method
SOILS/BUILDING MATERIALS[1]	
VOCs	CLP SOW SOM01.2
SVOCs	CLP SOW SOM01.2
PCBs	CLP SOW SOM01.2
Pesticides	CLP SOW SOM01.2
TAL metals (including mercury and cyanide)	CLP SOW ILM05.4
TCLP Metals	EPA SW-846 Method 1311 and CLP SOW ILM05.4
SPLP Metals	EPA SW-846 Method 1312 and CLP SOW ILM05.4
Asbestos	EPA 600/R-93-116
AIR	
Asbestos	NIOSH 7402
SURFACE WATER AND GROUNDWATER	
VOCs	CLP SOW SOM01.2
SVOCs	CLP SOW SOM01.2
PCBs	CLP SOW SOM01.2
Pesticides	CLP SOW SOM01.2
Filtered and Unfiltered TAL metals (except cyanide)	CLP SOW ILM05.4
Cyanide	CLP SOW ILM05.4
Total Hardness	EPA Method 130.1

Notes:

CLP	Contract Laboratory Program
PCB	Polychlorinated biphenyl
SVOC	Semivolatile organic compound
SOW	Statement of work
VOC	Volatile organic compound
TAL	Target Analyte List
TCLP	Toxicity characteristic leaching procedure
SPLP	Synthetic precipitation leaching procedure
EPA	U.S. Environmental Protection Agency
NIOSH	National Institute for Occupational Safety and Health

1 Building material analyses will be a "modified analysis" because the CLP laboratory will need to grind and homogenize oversized materials.
See Section 12.0 for complete citation of references for analytical methods (EPA 2005), (EPA 2006b), (EPA 2007b).

7.0 DECONTAMINATION PROCEDURES

During sampling activities, decontamination procedures will be followed by SulTRAC as outlined below. The DPT and hollow stem auger equipment will be steam cleaned before work begins and between sampling locations. To prevent cross contamination, measuring and sampling equipment will be decontaminated prior to the initiation of sample collection activities and between each two consecutive sampling locations. The equipment will be decontaminated following the general practices in SOP 002. A portable steam cleaner and an on-site source of potable water will be used for decontamination. All water derived from decontamination will be collected and temporarily stored in Department of Transportation (DOT)-approved, 55-gallon drums on site for characterization. Disposable sampling equipment will be used to collect individual samples only. Except for the detergent that will be used for the initial cleaning, the solutions used to decontaminate the field equipment will not be reused.

8.0 SAMPLE HANDLING PROCEDURES

SulTRAC will collect air, soil (includes soil, slag, sinter, debris pile, vegetation), building material, groundwater, and surface water samples; prepare the samples for shipment; complete all necessary documentation; and decontaminate non disposable equipment. Sample containers, preservatives, holding times, identification, documentation, COC, packaging, and shipping are discussed in this section.

8.1 SAMPLE CONTAINER, PRESERVATION, AND HOLDING TIMES

SulTRAC anticipates collecting air, soil, building material, groundwater, and surface water samples. Sample handling and procedures are different for each type of chemical group analysis and matrix type. These details are summarized in Table 8.

8.2 SAMPLE IDENTIFICATION

Samples will be identified by a unique sample identification number (see Table 9). The identifier will be comprised of the following information:

- Sample location (e.g. monitoring well identification number, MW-04)
- Other unique characteristic (see "Other sampling parameters" in Table 9)
- Sample date
- Sample type (field, field duplicate, or QA/QC)

Each sample will also be assigned an identifying number by CLP Forms-II Lite software. Forms-II Lite was developed to expedite sample documentation, track samples from the field to the lab, and reduce the most common documentation issues associated with sampling. The user will enter information regarding the site, project, sampling team, analysis, location, matrix, collection time/date, and sample and tag numbers, before or during the sample event. Because there are surveys to be completed prior to sampling of the piles and buildings, and the unknowns (previously discussed) associated with sampling depths at this site, SulTRAC will identify specific sample names after the start of the field campaign, once site surveys and preliminary direct- push technology results are logged.

TABLE 8
SAMPLE CONTAINER, PRESERVATION, AND HOLDING TIMES

Matrix	Analyte	Sample Container	Preservation Requirements	Maximum Holding Time (preparation/analysis)[1]
Air	Asbestos	25-mm diameter piece cassette loaded with a MCE filter or pore size 0.45mm. The filter should be backed by a 5-mm pore size MCE filter	NR	NR
Soil	VOCs	Three 40-mL glass containers with PTFE-lined septa and open open-top screw caps, pre-weighted and containing magnetic stir bars, and one container of sample filled with no headspace for determination of moisture content.	Iced 4 °C ± 2 °C	48 hours to preservation at laboratory/ 14 days for analysis following preservation
		At least three coring tools used as transport devices (e.g., 5-gram samplers), and one container of sample filled with no head space for determination of moisture content.	Frozen -7 °C to -15°C	48 hours (frozen) to preservation at laboratory for analysis after preservation
Soil	SVOCs	Two 4- or one 8-ounce wide-mouth glass jars	Cool to 4±2°C Immediately after collection	14 days/40 days
Soil	PCBs	Two 4- or one 8-ounce wide-mouth glass jars	Cool to 4 ±2°C Immediately after collection	14 days/30 days
Soil	Pesticides	Two 4- or one 8-ounce wide-mouth glass jars	Cool to 4±2°C Keep away from light	14 days/30 days
Soil	Metals (including Hg, CN))	Two 4- or one 8-ounce wide-mouth glass jars	Cool to 4°C ± 2°C immediately after collection	NA/6 months (Metals & Hg) 14 days/14 days (CN)
Soil	Asbestos	Two 4- or one 8-ounce wide-mouth glass jars	NR	180 days
Soil	TCLP Metals (except Hg)	One 8-ounce wide-mouth glass jar	Cool to 4 ±2°C immediately after collection	180 days to TCLP extraction/180 days to analysis
Soil	TCLP Mercury	One 4-ounce wide-mouth glass jar	Cool to 4 ±2°C immediately after collection	28 days to TCLP extraction/28 days to analysis
Soil	SPLP Metals (including Hg)	One 8-ounce wide-mouth glass jar	Cool to 4 ±2°C immediately after collection	180 days from collection to extraction/180 days from extraction to analysis 28 days for Hg

Matrix	Analyte	Sample Container	Preservation Requirements	Maximum Holding Time (preparation/analysis)[1]
Soil (bioavailability study)	Metals	3-gallon container	Cool to 4 ±2°C immediately after collection	14 days survival 28 days bioavailability
Soil (vegetation study)	Metals	1-gallon Ziplock-type bags	Cool to 4 ±2°C immediately after collection	NA/28 days if refrigerated 6 months if frozen
Soil (Bioavailability study)	Lead and arsenic	8-ounce wide-mouth glass jars	Cool to 4 ±2°C immediately after collection	NA/6 months
Building Material	VOCs	Granular Solids – Three 40-mL glass containers with PTFE-lined septa and open-top screw caps, pre-weighted and containing magnetic stir bars, and one container of sample filled with no headspace for determination of moisture content Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Iced to 4°C ±2°C	48 hours to preservation at laboratory 14 days from time of collection
Building Material	SVOCs	Granular Solids – Two 4- or one 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 ±2°C Immediately after collection	14 days/40 days
Building Material	PCBs	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4±2°C Immediately after collection	14 days/30 days
Building Material	Pesticides	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4±2°C Keep away from light	14 days/30 days

Matrix	Analyte	Sample Container	Preservation Requirements	Maximum Holding Time (preparation/analysis)[1]
Building Material	TAL metals (CN, Hg))	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Place sample in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 ±2°C immediately after collection	NA/6 months (Metals, Hg) 14 days/14 days (CN)
Building Material	Asbestos	Oversized concrete, wood, or stone samples – Place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample. Rubble: One 4-ounce, 8-ounce, or 16-ounce wide-mouth glass jar, depending on Size of matrix.	NR	NA/180 days
Building Material	TCLP Metals (including mercury and cyanide)	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Place sample in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 ±2°C immediately after collection	180 days to TCLP extraction/180 days to analysis
Water	VOCs	Three 40-mL glass vials with PTFE-lined septa and open-top screw caps	No headspace Cool to 4±2°C adjust pH to less than 2 with HCl	7 days/14 days
Water	SVOCs	Two 1-liter amber glass bottles fitted with PTFE-lined screw caps	Cool to 4±2°C Immediately after collection	7 days/40 days
Water	PCBs	Two 1-liter amber glass bottles, fitted with PTFE-lined screw caps	Cool to 4±2°C immediately after collection	7 days/40 days
Water	Pesticides	Two 1-liter amber glass bottles, fitted with PTFE-lined screw caps	Cool to 4±2°C immediately after collection	7 days/40 days
Water	TAL Metals (including mercury)	One 1-liter HDPE bottle with polyethylene-lined caps Dissolved metals sample: use a 0.45-µm size filter Particulate metals sample: no filter needed	HNO_3 to pH < 2 and cool to 4 °C (±2 °C) immediately after collection	NA/6 months for filtered (dissolved metals, Hg) and non filtered (metals, Hg)
Water	Cyanide	One 1-liter HDPE bottle with polyethylene-lined caps	NaOH to pH>12 and cool to 4±2°C immediately after collection	NA/14 days
Water	Total Hardness	500 mL HDPE bottle with polyethylene-line caps	HNO_3 to pH < 2 and cool to 4 °C (±2 °C) immediately after collection	NA/6 months

Notes:

µm	Micron
°C	Degrees Celsius
CLP	Contract Laboratory Program
CN	Cyanide
HCL	Hydrochloric acid
HDPE	High-density polyethylene
Hg	Mercury
HNO_3	Nitric acid
MCE	Mixed cellulose ester
mL	Milliliter
mm	Millimeter
NA	Not applicable
NaOH	Sodium hydroxide
NR	Not required
PCB	Polychlorinated biphenyl
PTFE	Polytetrafluoroethylene
SPLP	Synthetic precipitation leaching procedure
SVOC	Semivolatile organic compound
TCLP	Toxicity characteristic leaching procedure
VOC	Volatile organic compound

[1] Holding time is measured from time of sample collection to the time of sample extraction and analysis (EPA 2004).

TABLE 9
GENERALIZED SAMPLE IDENTIFICATION SCHEME

Matrix		Location Number	Other Sampling Parameters	Date	Example Identification	Notes
Soil Boring	SB	312 (3 digits)	surface (A) subsurface (B)	2008 (08) last two digits of year	SB312A-08 surface sample SB312B-08 subsurface sample	All sample locations will have new sample ID, in numerical order. Field sampler MUST indicate sinter, slag, soil, or debris pile in log and field notebook when sampling.
Soil Surface	SS	003 (3 digits)	Bioavailability Bioassessibility	2008 (08) last two digits of year	SS065-08	All sample locations will have new sample ID, in numerical order Field sampler MUST indicate sinter, slag, soil, or debris pile in log and field notebook when sampling.
XRF-Screened Surface Soil from Gridded Polygons	n/a	poly_1, poly_2, poly_3, poly_4, poly_5, poly_6	001(3 digits) number of samples in each polygon	n/a	poly_1_036	Each polygon will have a pre-determined number of screening/ sampling locations for XRF. Once a correlation is performed on the XRF data, approximately 50 samples will be sent to CLP laboratory for analysis Field sampler MUST indicate sinter, slag, soil, or debris pile in log and field notebook when sampling.
Building Material	BM	026 (3 digits)	Wood (W) Stone (T) Brick (K) Concrete (C) Other (Z)	2008 (08) last two digits of year	BM026W-08 wood sample BM032K-08 brick sample BM101Z-08 other sample (e.g. plastic) BM087T-09 stone sample	All sample locations will have new sample ID, in numerical order
Surface Water	SW	010 (3 digits)	n/a	May 2008 (0508) two-digit month and year	SW010-0508	Same sampled locations will have same sample location number with different date, in sample ID SW010-0508 sampled in May 2008 SW010-0808 sampled in Aug. 2008

Matrix		Location Number	Other Sampling Parameters	Date	Example Identification	Notes
Groundwater	MW	19 (no digit requirement)	n/a	May 2008 (0508) two-digit month and year	MW19-0508	Same sampled locations will have same sample location number with different date, in sample ID MW19-0608 sampled in June 2008 MW19-0908 sampled in Sep. 2008

Notes:
All Sample IDs and associated sampling information are included in Table 3

BM	Building material
CLP	Contract Laboratory Program
MW	Monitoring well
n/a	Not applicable
SB	Soil Boring
SS	Soil surface sample
SW	Surface water
XRF	X-ray fluorescence

8.3 SAMPLE LABELS

Forms-II Lite generates labels for each sample. A sample label will be affixed to all sample containers. The label will be completed with the following information:

- Project number
- CLP case number
- CLP sample number
- Sample station name (this is the sample identity [ID] discussed above)
- Sample collection date and time
- Preservative
- Sample collector's initials
- Analysis
- Sample tag number.

After labeling, if required, each sample will be will be preserved as required (see Table 8).

8.4 SAMPLE DOCUMENTATION

Sampling activities will be documented in a logbook using a ballpoint pen. The header of each page shall include the site location name, date, and project number. At the start of each day, the weather, site condition, field staff present, subcontractors present, and any conducted safety meeting or other, shall be noted. The collection time, sample identification number (not CLP ID), sample depth (if appropriate), sample location description, field observations, sampler's name, time of sample collection, and analyses will be recorded in the logbook for each and every sample. For soil samples, the delegation of slag, sinter, soil, or debris pile will be entered. For building samples, the type of material must be entered. Every MS/MSD and duplicate should be clearly designated in the fieldbook. Collection of rinsate samples and preparation of trip blanks should be documented with applicable parameters in the same manner as described above.

Each page of the logbook will be dated, numbered (if appropriate), and signed at the bottom by SulTRAC personnel. Any residual space on the last page of each day's log book shall be crossed out with a single line. Each new sampling day shall begin on a new page in the log book. Any corrections made during the same day of sampling should be crossed out with one single line, or the term "backnote" can be inserted to account for missed time.

The field team leader oversees that all documentation in the logbook is done appropriately and accordingly and should check this daily. Any corrections or additions can be made on a subsequent page with appropriate documentation, although this is not recommended and corrections or additions are at best made on the same day as sampling.

All field log books must be kept secure at all times by the field team leader while conducting field work. As possible, all field log books shall be scanned electronically at high resolution, minimum 300 x 300. If electronic scans can not be conducted after one week of continuous field work, high-resolution hardcopies must be made and kept secure until electronic scanning can be performed. All completed field books and any hardcopies will be stored with the project manager in the Chicago Office. Field data records will be maintained in accordance with Multi-Media Investigation Manual and Procedures (EPA 1992) and SulTRAC's FSP.

8.5 SAMPLE CHAIN OF CUSTODY

SulTRAC will use standard sample custody procedures to maintain and document sample integrity during collection, transportation, storage, and analysis in accordance with the SulTRAC RAC II Contract Level QAPP. A sample will be considered in custody if one of the following statements applies:

- It is in a person's physical possession or view.
- It is in a secure area with restricted access.
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Forms-II Lite generates and prints COC forms, called traffic reports, in Forms II Lite (a laboratory copy and a region copy). The laboratory copy will be sealed inside the lid of the cooler. COC procedures provide an accurate written record that traces the possession of individual samples from the time of collection in the field to the time of acceptance at the laboratory. One COC record will be generated for

each cooler shipped. The COC record also will be used to document all samples collected and the analysis requested. The following information will be documented on the COC form:

- Project name and number (region copy only)
- CLP case number
- CLP sample numbers
- Sample tag numbers
- Sampling location (station identification)
- Name and signature of sampler
- Destination of samples (laboratory name)
- Sample ID number
- Date and time of collection
- Number and type of containers filled
- Analysis requested
- Preservatives used (if applicable)
- Sample designation (grab or composite)
- Special instructions (e.g., laboratory needs to sub-sample oversized material or perform additional homogenization)
- Signatures of all samplers
- Signatures of individuals involved in custody transfer, including the date and time of transfer
- Airbill number (if applicable)
- Project contact and phone number
- Custody seal number

SulTRAC will follow the procedures in the EPA Region 5 CRL "Superfund Amendments and Reauthorization Act (SARA)/Superfund Sample Handling Manual" (EPA 1989) to complete the documentation listed above.

SulTRAC will appoint one of its field technical staff members to serve as the sample custodian. Upon completion of all required documents, the sample custodian will sign and date the document and list the time of the sample collection. The custodian will also confirm the completeness of all descriptive information on the COC forms, which will be included with each shipping container. Two custody seals total will be used: one with the custody seal placed across the latch of the container and the other placed on the opposite side of the container lid. The lid will be securely taped shut for shipment. The field sample custodian shall send the original copies of the COC region copy to the project manager, who in turn will submit these to the Region 5 Sample Management Office (SMO), care of Warren Layne, within 5 working days. The sample custodian will also retain and scan all copies of all COCs (laboratory and region) for the project files.

8.6 SAMPLE PACKING AND SHIPPING

The following procedures will be implemented when samples collected during this project are shipped:

- All samples jars will be individually wrapped with bubble wrap or other packing material and placed in their own individual ziplock-type bags. Each sample will have its CLP ID tag accompanying the sample package.
- Ice will be double bagged in large ziplock-type bags and placed at the bottom of the cooler. If the cooler has a drain, it will be taped shut both inside and outside of the cooler.
- The cooler will be lined with bubble wrap or other packing material, and all individually packaged samples will be placed into one large plastic bag and tied after all sample jars have been input. Sufficient packing material will be used to prevent sample containers from breaking during shipment.
- Additional ice, double bagged, will be added on top of the tied plastic bag full of samples. Enough ice will be added to maintain a sample temperature of $4 \pm 2°C$. SulTRAC shall prepare, label, and place a temperature blank in each cooler.
- Notify the laboratory if a sampler suspects that any sample contains anomalously high or low concentrations (handwrite this anomaly directly on laboratory copy of COC), or if there may be a sampled substance that would require laboratory personnel to take safety precautions.

- The COC, specific to each cooler, will be sealed inside a plastic bag, and taped to the inside of the cooler lid. Ensure that the COC is signed by all samplers and the custody seal numbers are included on the COC. Include with the COC a return pre-paid air bill so the cooler may be returned to SulTRAC.
- The cooler will be closed and taped shut with strapping tape around both ends.
- Signed and dated custody seals will be placed on the front and side of each cooler. Wide clear tape will be placed over the seals to prevent accidental tearing.
- The air bill, if required, will be completed before the samples are relinquished to the carrier.
- The COC will be transported within the taped sealed cooler. When the cooler is received at the analytical laboratory, laboratory personnel will open the cooler and sign the COCs to document transfer of samples.
- The Superfund SMO will be notified if the laboratory should expect to receive samples on a Saturday. SulTRAC should call its CLP sample coordinator who in turn will notify the SMO.

All shipping containers will be labeled as required by the DOT. After packaging, the samples will be shipped to the CLP laboratory specified by the EPA Region 5 Regional Sample Control Coordinator.

9.0 DISPOSAL OF INVESTIGATION-DERIVED WASTE

IDW is waste generated from an activity related to determining the nature and extent of contamination at OU2. It includes solid and any hazardous waste, media (e.g. soil, groundwater, surface water), and debris (e.g. building materials, debris piles) that contain "listed" hazardous waste or that exhibit a characteristic of a hazardous waste. It includes media and debris that is not hazardous, but is contaminated with hazardous constituents.

IDW generated during the field sampling activities at M&H Site includes homogenized soil extracted by borings and monitoring well installation, purge water from well development and groundwater sampling, as well as wastewater from decontamination and equipment rinsate procedures. Soil, groundwater, and wastewater will be containerized in separate 55-gallon drums. Each drum will be clearly marked to indicate the date of collection, its waste contents, and other generator information. Each drum, prior to DOT classification, will have a completed "WASTE MATERIAL" label affixed to on the exterior side. This label will include site, address, contents, boring or well depth, operation, accumulation date, and consultant phone number information. All information must and will be completed for each drum. Prior to off-site disposal, the drums will be relabeled with appropriate DOT identification and classification information.

All IDW will be disposed of as required by state and local regulations following receipt of results for IDW soil and water analysis. Additional IDW generated as a result of soil sampling will include disposable personal protective equipment (PPE) and sampling equipment. Disposable PPE and sampling equipment will be managed according to the level of contamination encountered during field activities. In general, PPE will be managed as non hazardous solid waste, particularly if little contact occurs with the sampling media and low levels of contaminants are involved. Therefore, this waste will be double bagged and disposed of with municipal trash. Additional information concerning IDW and site-specific disposal requirements can be found in the Site Management Plans (SulTRAC 2007b).

10.0 HEALTH AND SAFETY PROCEDURES

All field activities will be conducted in accordance with SulTRAC Health and Safety Plan (HASP), which is among the site-specific plans prepared for the WA (SulTRAC 2008c). Prior to initiation of field activities, all SulTRAC field personnel and subcontractors will read and sign the HASP, indicating that they understand the plan and agree to operate in accordance with its requirements. All SulTRAC personnel and subcontractors must have 40-hour hazardous waste and emergency response training, and proof of certification must be filed with the signed HASP. A complete copy of the site-specific plans, including the updated Phase II HASP, will be maintained by the field sampling team at the site.

11.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

All QA activities will be conducted in accordance with the SAP. A copy of the SAP will be maintained by the field sampling team for immediate use in resolving any QA issues that might arise during field activities.

QC samples will be collected at the following frequencies:

- Field Duplicate: One per 10 environmental samples will be collected, with a minimum of one per sample matrix.
- Trip Blank Samples: One trip blank will be included in each cooler containing samples for VOC analysis.
- MS/MSD Samples: One per 20 environmental samples per matrix will be collected.

Field duplicate samples consist of two separate samples collected from the same sampling location and depth, using the same equipment and sampling procedures. A trip blank is a clean sample of a matrix that is taken from the laboratory to the sampling site and transported back to the laboratory, without having been exposed to sampling procedures (typically analyzed only for volatile compounds). This sample is not to be labeled or identified as a trip blank for the CLP laboratory. MS/MSD is an environmental sample divided into two separate aliquots, each of which is spiked with known concentrations of target aliquots. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. For groundwater samples, the MS/MSD requires collecting triple sample volume (three sets of vials), while for solid matrices, the MS/MSD does not require extra volume collection. All samples should be identified as MS/MSD for the CLP laboratory.

12.0 REFERENCES

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FIGURES

(Nine Sheets)

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

FIGURE 1
SITE LOCATION MAP

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

ST SulTRAC

NORTH AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB421	21
SB422	22
SB423	23
SB424	24
SB425	25
SB426	26
SB427	27
SB428	28
SB429	29
SB430	30

NEAR BLDG 100 (PCB AREA)	
SOIL BORING ID	MAP ID# & SYMBOL
SB401	1
SB402	2
SB403	3
SB404	4
SB405	5
SB406	6
SB407	7
SB408	8
SB409	9
SB410	10

NORTHWEST OF ROLLING MILL	
SB411	11
SB412	12
SB413	13
SB414	14
SB415	15
SB416	16
SB417	17
SB418	18
SB419	19
SB420	20

NORTHEAST PERIPHERY AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB441	41
SB442	42
SB443	43
SB444	44
SB445	45
SB446	46
SB447	47
SB448	48
SB449	49
SB450	50

INSIDE ROLLING MILL	
SOIL BORING ID	MAP ID# & SYMBOL
SB431	31
SB432	32
SB433	33
SB434	34
SB435	35
SB436	36
SB437	37
SB438	38
SB439	39
SB440	40

MAIN PLANT AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB451	51
SB452	52
SB453	53
SB454	54
SB455	55
SB456	56
SB457	57
SB458	58
SB459	59
SB460	60

ACID RESERVOIRS
ROLLING MILL (DRY STORAGE)
BUILDING 1943 (DRY STORAGE)
FURNACES
OXIDE PLANT
CONCENTRATION PLANT
POTTERY
LEACHING PLANT
BOILER HOUSE
MOTOR FLOOR PUMP BASES (40')
AST

LEGEND:

OPERABLE UNIT OU2 SITE BOUNDARY

ROLLING MILL HISTORIC STRUCTURE (APPROXIMATE LOCATION) (NOT ALL HISTORIC STRUCTURES ARE OUTLINED)

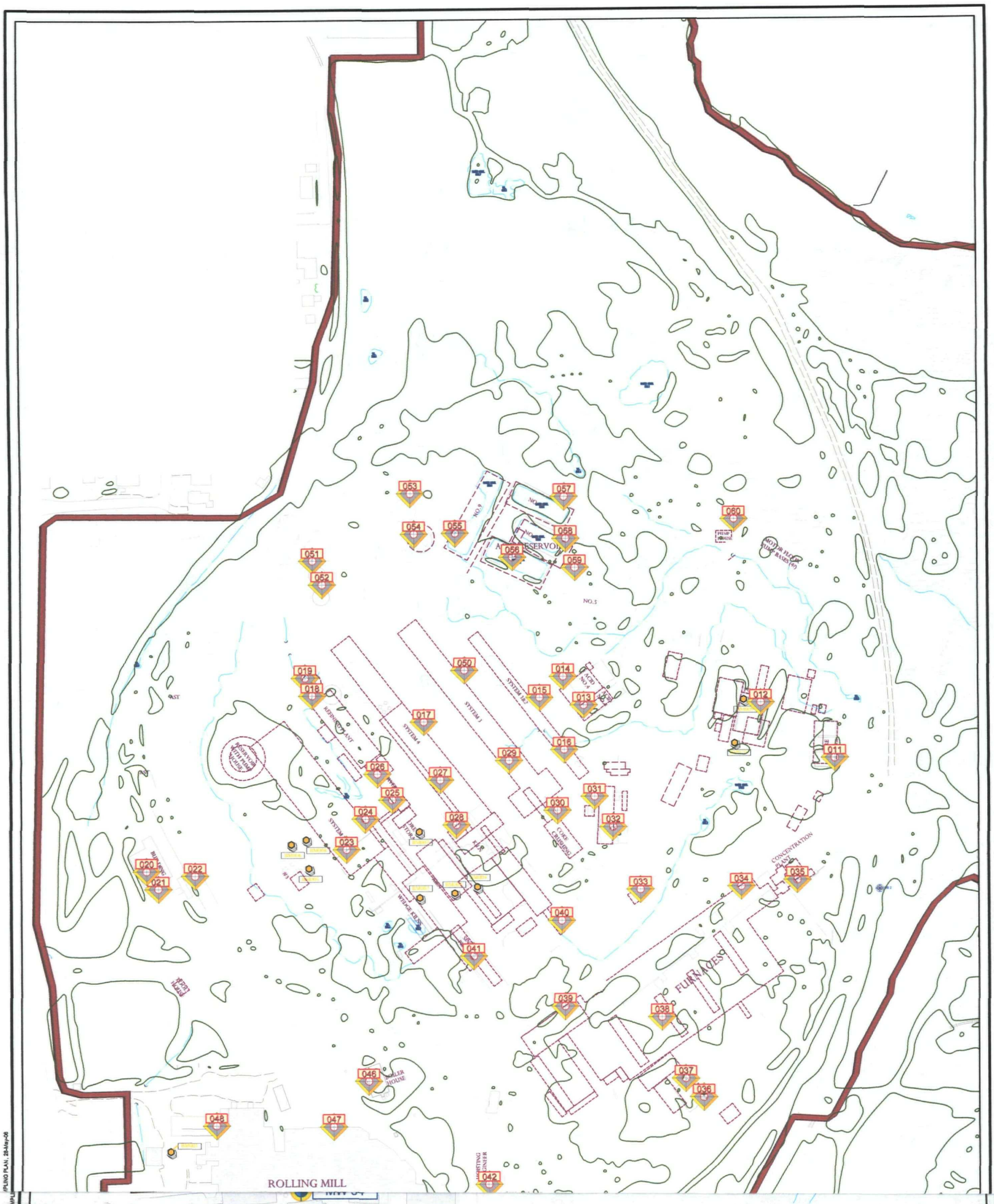
N

GRAPHIC SCALE
100 0 50 100 200 400
(IN FEET)
1 INCH = 150 FEET

SulTRAC

MATTHIESSEN & HEGELER ZINC COMPANY SITE
STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS
EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

FIELD SAMPLING PLAN
FIGURE 2
PROPOSED SOIL BORING LOCATION MAP



GRAPHIC SCALE

125 0 125 250 500

(IN FEET)

1 INCH = 250 FEET

N

LEGEND:

- MW 34 — PROPOSED MONITORING WELL LOCATION (17)
- P6 — PROPOSED LOCATION OF 2-INCH DIAMETER PIEZOMETER (6)
- DB MW 35 — PROPOSED DEEP BORING (3)*
- MW 10 — EXISTING GROUNDWATER MONITORING WELL (17)
- MW 28 / MW 20 — EXISTING GROUNDWATER MONITORING WELL CLUSTER - DEEP AND SHALLOW SCREENS (1 GROUP OF 2 WELLS)

NOTES:

* BACKFILLED PRIOR TO INSTALLATION OF PIEZOMETER P1 AND MONITORING WELLS MW 25 AND MW 35

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

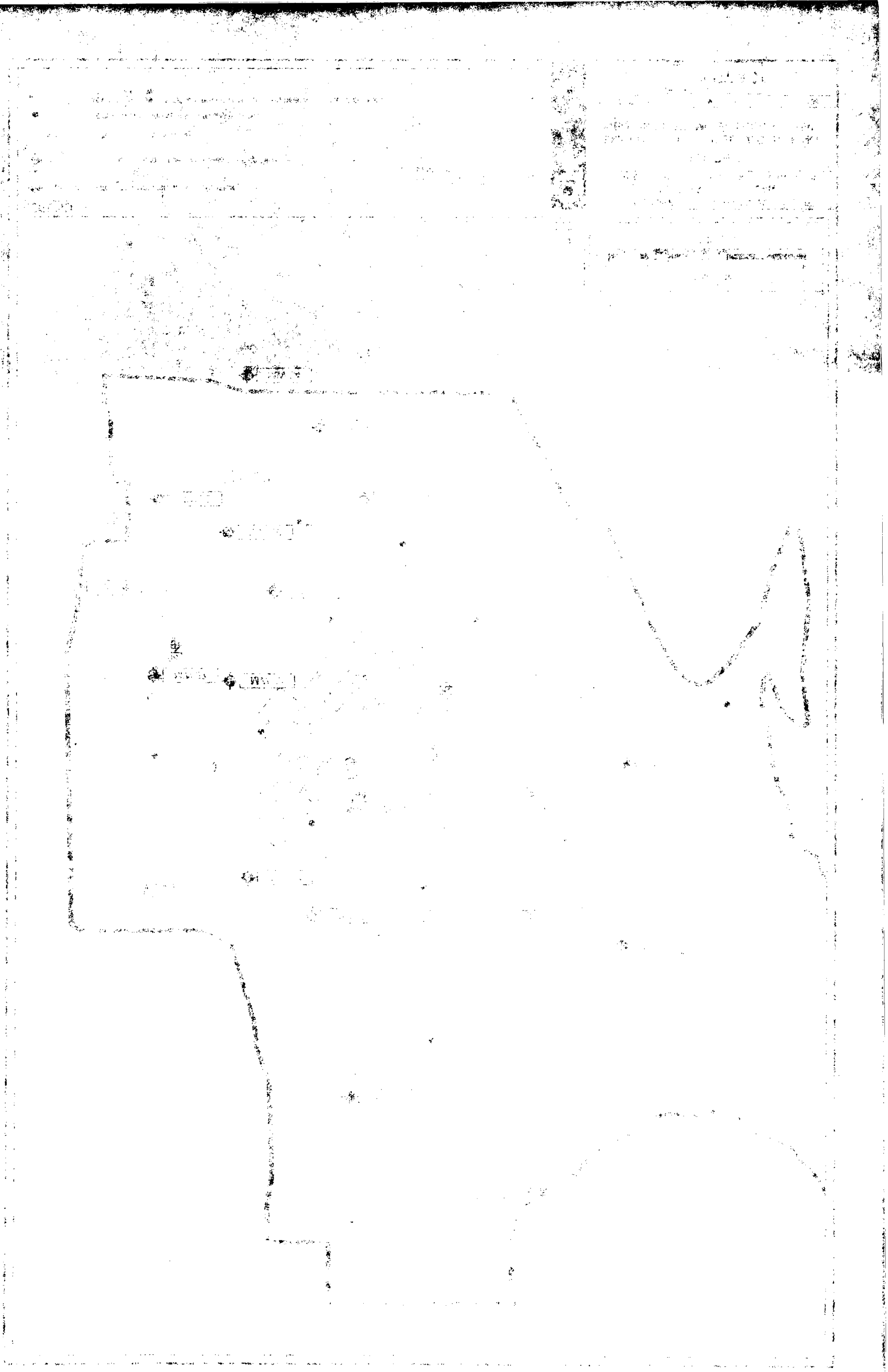
FIGURE 4

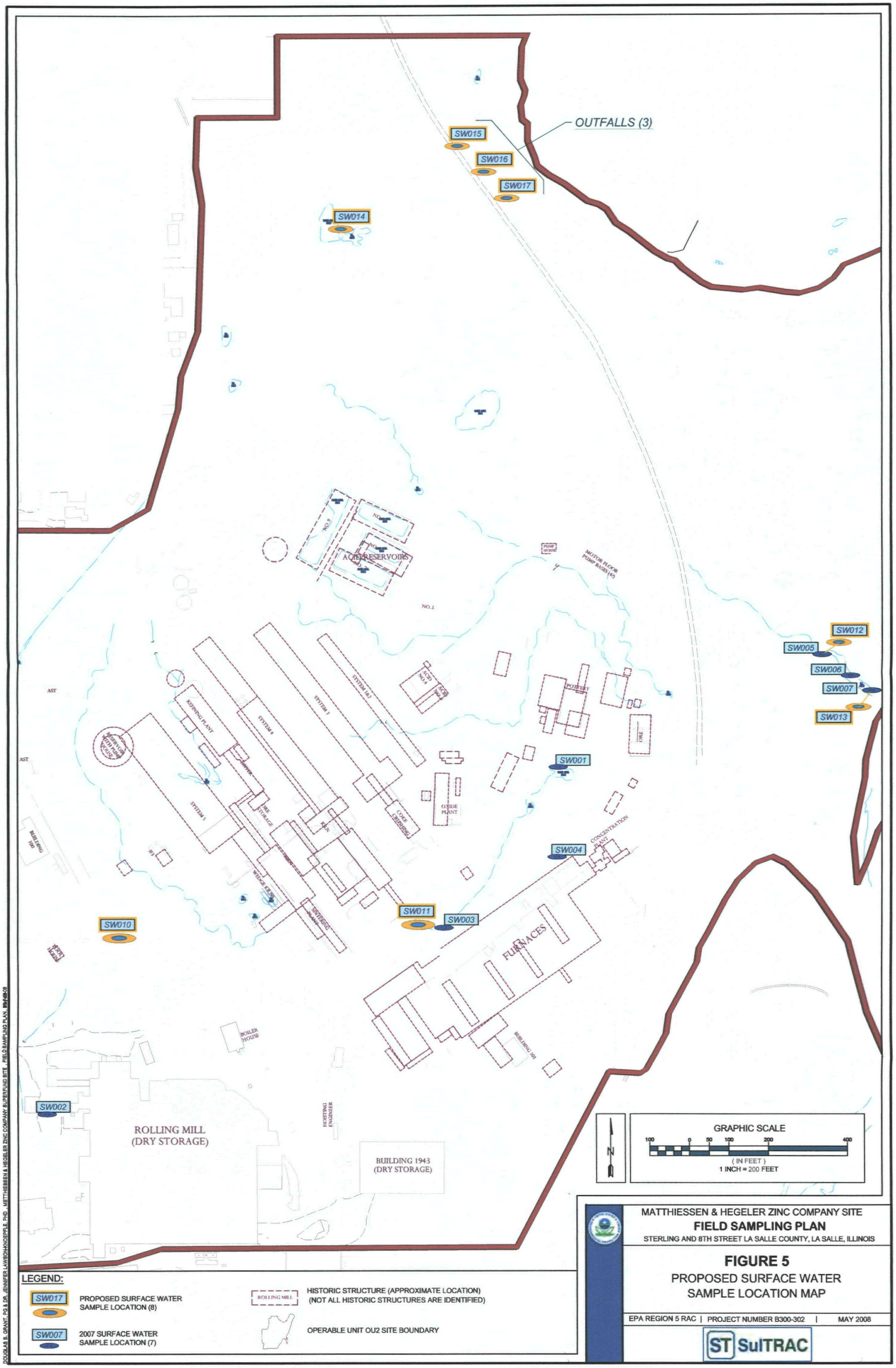
EXISTING AND PROPOSED MONITORING WELL AND PIEZOMETER LOCATION MAP

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

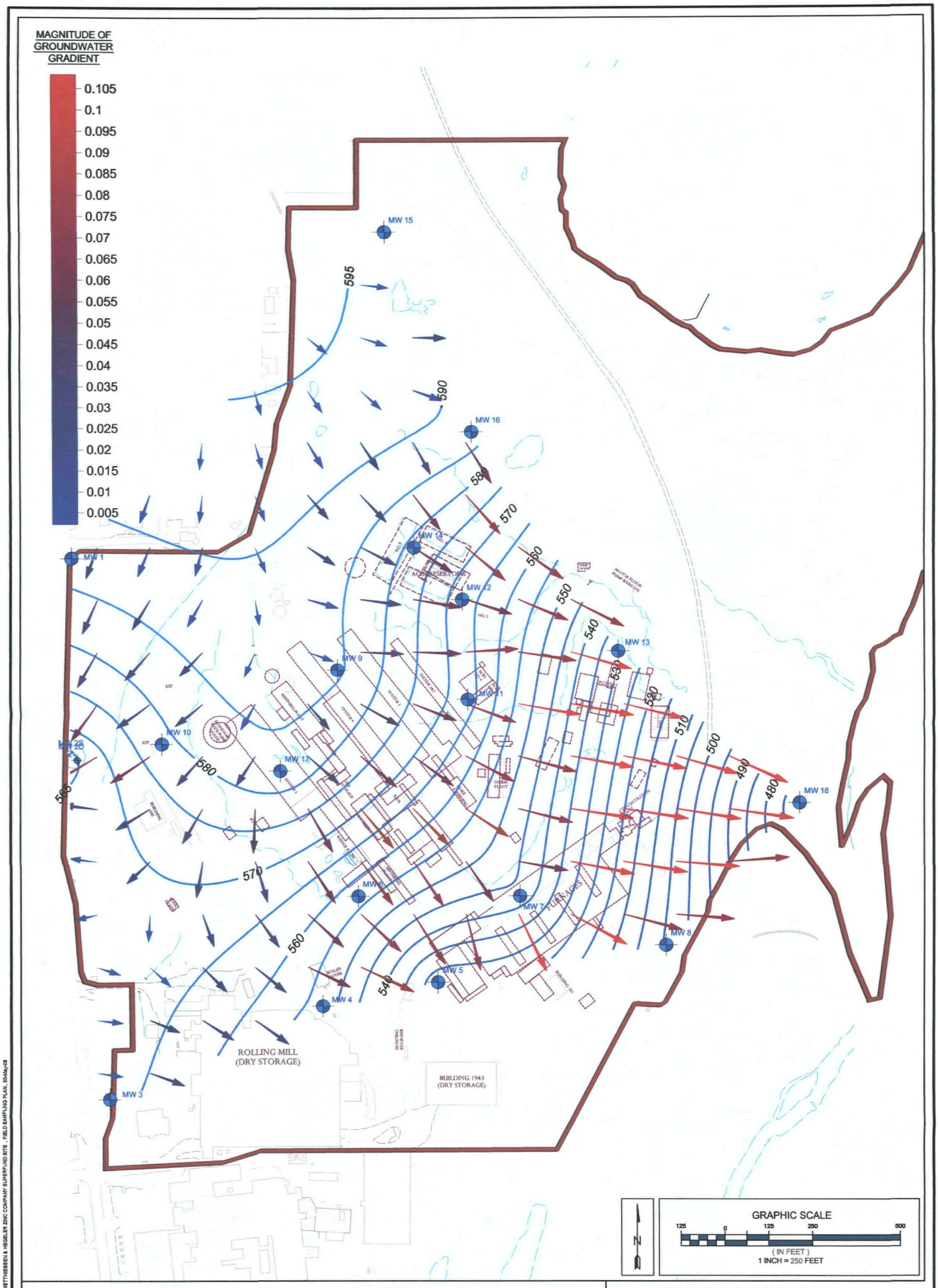
ST SulTRAC

DOUGLAS S. GRANT, PG & DR. JENNIFER LAVSCHAKVOEPPLE, PHD - METTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE - FIELD SAMPLING PLAN, 28-May-08





DOUGLAS B. GRANT, PG & DR. JENNIFER LAWSON-KNOEPPLE, PhD. - MATTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE - FIELD SAMPLING PLAN, 05/08/08



LEGEND:

- GROUNDWATER MONITORING WELL
- GROUNDWATER MONITORING WELL CLUSTER (DEEP AND SHALLOW SCREENS)
- GROUNDWATER ELEVATION ISOCONTOURS (FEET MEAN SEA LEVEL)
- GROUNDWATER GRADIENT *MAGNITUDE INDICATED BY VECTOR LENGTH AND COLOR*

NOTES:

1. GROUNDWATER ELEVATION DATA COLLECTED IN NOVEMBER 2007.

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

FIGURE 6

GROUNDWATER POTENTIOMETRIC MAP: NOVEMBER 2007

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

SulTRAC

DOUGLAS S. GRANT, PG & DR. JENNIFER LAW/BONHOEFFLE, PHD. ,METTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE , FIELD SAMPLING PLAN, 30-May-08

MAGNITUDE OF GROUNDWATER GRADIENT

0.105
0.1
0.095
0.09
0.085
0.08
0.075
0.07
0.065
0.06
0.055
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005

MW 1
MW 2S
MW 2D
MW 3
MW 4
MW 5
MW 6
MW 7
MW 8
MW 9
MW 10
MW 11
MW 12
MW 13
MW 15
MW 16
MW 17
MW 18

590
580
570
560
550
540
530
520
510
500

ROLLING MILL (DRY STORAGE)
BUILDING 1943 (DRY STORAGE)
FURNACES
CORE RESERVOIRS
SETTLING PLANT
CORE PLANT
CORE TREATMENT
CONCENTRATION
MOTOR FLOOR PUMP ROOM (#7)
HOISTING ENGINEER
BUILDING 101
BUILDING 1300
AST

N

GRAPHIC SCALE
125 0 125 250 500
(IN FEET)
1 INCH = 250 FEET

LEGEND:

MW 16 — GROUNDWATER MONITORING WELL

MW 2S / MW 2D — GROUNDWATER MONITORING WELL CLUSTER (DEEP AND SHALLOW SCREENS)

GROUNDWATER ELEVATION ISOCONTOURS IN FEET MEAN SEA LEVEL. DARKER INDICATES LOWER ELEVATION

GROUNDWATER GRADIENT
MAGNITUDE INDICATED BY VECTOR LENGTH AND COLOR. MARCH 2008 VECTORS NON-SHADED.

NOTES:
1. GROUNDWATER ELEVATION DATA COLLECTED IN MARCH 2008.

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

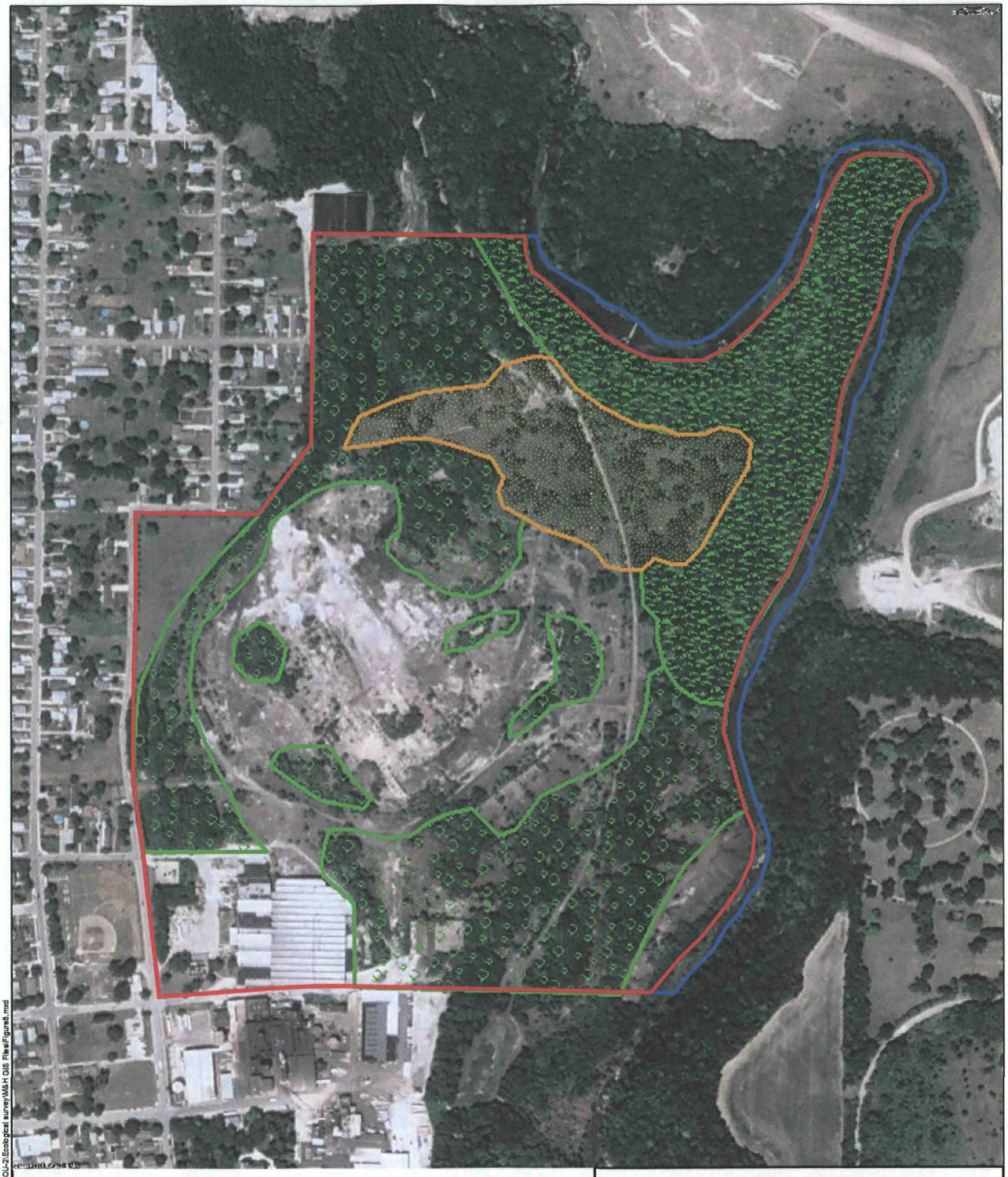
FIGURE 7

GROUNDWATER POTENTIOMETRIC MAP: MARCH 2008

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

SulTRAC

DOUGLAS B. GRANT, PG & DR. JENNIFER LAWSON-KNOEPPLE, PHD , METTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE , FIELD SAMPLING PLAN , 30-May-08



Legend

- Disturbed Woodland-Grassland
- Oak-Hickory Woodland
- Savannah
- Riverine
- OU2 Boundary

N W E S

0 250 500

Feet

Source: 2006 USDA National Agriculture Imagery Program, LaSalle County, Illinois

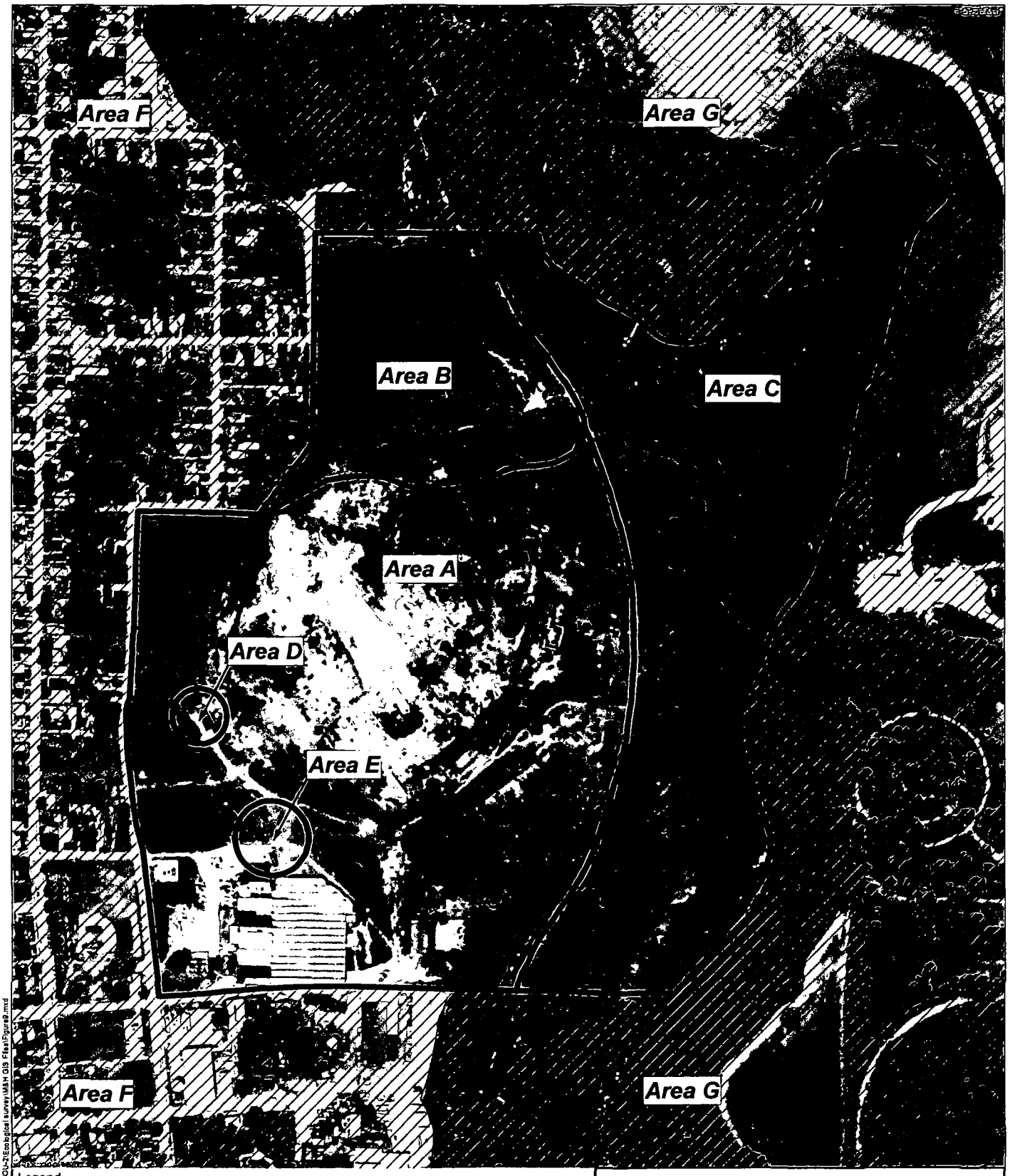
M&H Ecological Evaluation
LaSalle, Illinois

Figure 8
OU2 Habitat Types

ST SulTRAC

Date: 05/28/08 Drawn By: Bill Spiking Project No: G9021016.103

S:\PROJECTS\RAC Region5\Matthiessen & Hegeler Zinc\OU-2\Ecological survey\M&H GIS Files\Figure8.mxd



Legend

Approximate Off-site Exposure Areas

Appoximate On-site Exposure Areas

Area A : OU2 main industrial plant area
Area B : Wooded area north of main industrial area
Area C : Wooded area in the northeast periphery
Area D : PCB area near Building 100
Area E : TCE area northwest of rolling mill
Area F : Residential areas west of OU2
Area G : Area downwind and east of the Little Vermilion River

N
W E
S

0 250 500
Feet

Source: 2006 USDA National Agriculture Imagery Program, LaSalle County, Illinois

M&H Ecological Evaluation
LaSalle, Illinois

Figure 9
OU2 Exposure Areas

ST SulTRAC

Date: 05/28/08 Drawn By: Bill Spilding Project No: G9021016.103

S:\PROJECTS\RAC Region5\Matthiessen & Hegeler Zinc\OU-2\Ecological survey\M&H GIS Files\Figure9.mxd

STANDARD OPERATING PROCEDURES

(357 Sheets)

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

AIR QUALITY MONITORING

SOP NO. 073

REVISION NO. 1

Last Reviewed: November 1999

Quality Assurance Approved

May 26, 1993

Date

1.0 BACKGROUND

Air quality monitoring is performed to evaluate materials in the air from the site. Particulates, volatile organic compounds (VOC), and semivolatile organic compounds (SVOC) in the air can present potential health risks around the site. This standard operating procedure (SOP) establishes the requirements and procedures for air quality monitoring. This section discusses the purpose and scope of the SOP and lists the requirements and resources needed to monitor air quality. Section 2 outlines the procedures to use when collecting air quality samples using different types of instruments.

1.1 PURPOSE

This SOP establishes the requirements and procedures for monitoring air quality.

1.2 SCOPE

This SOP provides only a broad overview of recommendations for monitoring air quality. This SOP is to be used in conjunction with U.S. Environmental Protection Agency (EPA) guidance on air quality monitoring and the instruction manuals included with the sampling equipment.

This SOP also provides general information on air sampling techniques and equipment, sample locations, criteria for initiating sampling, and analytical procedures for airborne particulates, volatile organic compounds (VOC), and semivolatile organic compounds (SVOC).

Those using this SOP should be familiar with EPA analytical methods 608, TO-1, TO-2, TO-3, TO-4, TO-10, and TO-14.

1.3 DEFINITIONS

None

1.4 REFERENCES

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EPA. 1988. *Compendium Method TO-14, The Determination of Volatile Organic Compounds (VOC) in Ambient Air Using SUMMA Passivated Canister Sampling and Gas Chromatographic Analysis.* Quality Assurance Division. Research Triangle Park, North Carolina.

1.5 REQUIREMENTS AND RESOURCES

Depending on the type of air quality sampling to be conducted, some or all of the following equipment will be required:

- A General Metal Works Model PS-1 High Volume Sampler® is needed to collect samples of airborne particulates and SVOCs.
- A Spectrex Model PAS-3000 Personal Air Sampler® with a carbon molecular sieve (CMS) cartridge or a Tenax gas chromatograph (GC) adsorbent cartridge may be used to collect samples of VOCs.
- A SUMMA® canister may be used to collect samples of VOCs.
- A Gelman GN® filter is needed to collect samples of asbestos fibers.

2.0 PROCEDURES

This section discusses the procedures to use when collecting various types of air quality samples. This section also discusses procedures for identifying sampling locations, criteria for initiating sampling, analytical procedures for air sample analysis, and quality control (QC) procedures.

2.1 SAMPLING TECHNIQUES AND INSTRUMENTATION

This section presents information on sampling techniques and equipment used for sampling airborne particulates, SVOCs, VOCs, and asbestos. More detailed information about each instrument, including instrument calibration procedures, can be found in the instrument operating manuals, which are maintained with each instrument.

2.1.1 Particulates and Semivolatile Organic Compounds

A General Metal Works Model PS-1 High Volume Sampler® (PS-1 sampler) is used to collect samples of airborne particulates and SVOCs, including pesticides and polychlorinated biphenyls (PCB). This sampling technique uses a battery-driven pump to draw air through a filter and a polyurethane foam (PUF) plug cartridge. Contaminants in the air adhere to the filter and the PUF plug cartridge. The filter and cartridge are then submitted to an analytical laboratory where they are analyzed for the contaminants of concern.

The pump uses a bypass blower motor equipped with an independent cooling fan to sample at rates of up to 280 liters per minute. Power is provided by two rechargeable 12-volt batteries connected in series to provide a 24-volt power source. In this configuration, the pump can operate at full power for more than 4 hours. The PS-1 sampler equipment is housed in an 18.75- by 18.75- by 52.5-inch anodized aluminum shelter. In the upper portion of the shelter, a dual-chambered sampling module contains both the filter and the PUF plug cartridge.

Sampled air first moves through the upper portion of the sampling module. Incoming air passes through a 4-inch-diameter Teflon® filter that collects airborne particulates. The PS-1 sampler's range of operational

flow rates and housing design favor the collection of particulates with diameters between 0.1 and 100 microns. A collection efficiency of 99 percent can be obtained for particulates with a 0.3-micron diameter.

After passing through the filter, air enters the lower portion of the PS-1 sampler where it passes through a 3-inch-long, cylindrical glass cartridge containing the PUF plug cartridge. The PUF plug cartridge adsorbs SVOCs, pesticides, and PCBs. The sampling efficiencies of the PUF plug cartridge for various compounds are provided in EPA analytical method TO-4.

Airborne particulates also can be sampled using two other types of instruments: a total suspended particulate meter and a PM-10 sampler. These instruments can be used to capture particulates less than 10 microns in diameter. Both of these samplers use a battery-driven pump to draw air through a filter to capture the particulates. The filter is submitted to an analytical laboratory where it is weighed to determine particulate levels.

2.1.2 Volatile Organic Compounds and Asbestos

A Spectrex Model PAS-3000 Personal Air sampler® (PAS-3000 sampler) can be used to sample VOCs and asbestos. The PAS-3000 sampler draws air through an asbestos filter and a CMS cartridge or a Tenax GC cartridge. The PAS-3000 sampler operates using a series of eight 1.25-volt rechargeable nickel-cadmium batteries connected in series. The maximum flow rate through the PAS-3000 sampler is 500 milliliters per minute. The PAS-3000 sampler can operate continuously for 6 to 10 hours before its batteries need to be replaced or recharged.

Air is first drawn through a filter that captures asbestos fibers. Asbestos fibers are collected using a 25-millimeter-diameter Gelman GN® filter. This filter is made from mixed cellulose esters and has a pore size of 0.8 micron.

Air is then drawn through the CMS cartridge or Tenax GC cartridge. The CMS cartridge used in the PAS-3000 sampler is a Model 300 Supelco Carbotrap. This stainless-steel cartridge is filled with three specialized adsorbents: Carbotrap C, Carbotrap, and Carbosieve S-III®. Glass wool plugs separate the adsorbent materials and are packed into the ends of the cartridge. The CMS cartridge is specifically

designed to efficiently adsorb and desorb all VOCs listed in EPA analytical methods TO-1, TO-2, and TO-3, whether present individually or in complex mixtures.

A Tenax GC cartridge also can be used in the PAS-3000 sampler. Tenax GC is an adsorbent that traps VOCs. A stainless-steel tube is filled with the Tenax material, and air is then drawn through the tube.

Another method that can be used to sample VOCs is the SUMMA canister. This sampling technique collects samples by drawing air into an evacuated stainless-steel canister that has been specially treated to eliminate active adsorption sites. If desired, a pump and mass flow controller may be used to fill the canister slowly over an extended period.

2.2 SAMPLING LOCATIONS

To estimate the impact of contamination on air quality, air sampling should be conducted both upwind and downwind of the suspected contamination source. Upwind and downwind sample locations must be selected through an evaluation of the predominant wind direction in the area to be sampled. The predominant wind direction must be determined by analyzing data from nearby wind monitoring stations. Because the predominant wind direction can vary on a seasonal basis, both the annual and seasonal characteristics of the wind must be considered.

Wind monitoring stations are often located at airports or at other stations maintained by the National Weather Service. If an established wind monitoring station cannot be located near the site to be sampled, a temporary wind monitoring station should be established at the site.

Wind monitoring also should be conducted during air sampling. While air sampling is being conducted, winds that blow from the suspected contaminant source into the selected downwind sector must occur frequently. These winds must typically persist for several hours during the sampling event to allow a multi-hour sampling run to be completed.

Because wind direction may vary considerably during a period of several hours, it is generally preferable to use at least three or four downwind air monitoring sites simultaneously. These monitoring sites should be

located a sufficient distance apart so that the sample collected from at least one site will be representative of the true air quality, even if a slight shift from the optimal wind direction occurs.

Air quality samplers should be located in unobstructed areas at least 2 meters from any obstacle to air flow. The exhaust hose of each sampler should be stretched out downwind of the sampler's intake port to prevent any recycling of air.

2.3 CRITERIA FOR INITIATING SAMPLING

The decision to initiate sampling should be made only after carefully analyzing meteorological conditions. The meteorological conditions that are required before initiating sampling include the following:

- Winds from a selected direction sector that will produce net transport from the waste site toward the downwind air quality monitors and a forecast that these winds will persist throughout the sampling event
- Atmospheric stabilities in the neutral to stable range; moderately unstable conditions also acceptable for summer sampling events
- No precipitation

Air quality samples can be collected as discrete grab samples. However, samples are generally collected continuously over a period of several hours, and a minimum sampling time of 2 hours is usually desirable. The exact duration of the sampling will be based on the meteorological conditions, the requirements of the sampling equipment, and the individual project objectives.

2.4 EXPERIMENTAL PROCEDURE AND ANALYSIS

This section details the protocols and procedures for collecting, handling, and analyzing air quality samples. A sampling event can range from collecting a single grab sample to continuous sampling over a 24-hour period depending on meteorological conditions, instrument performance, and project objectives. After sampling is completed, the filters and cartridges from the samplers will be collected. All samples will be placed in clean containers, sealed from contact with outside air, and clearly labeled with their sample

location and the date and time that sampling was conducted. The samples will then be sent to an analytical laboratory for analysis using the chain-of-custody procedures.

2.4.1 Particulate and Semivolatile Organic Compounds

The PS-1 sampler will be operated at a rate of approximately 280 liters per minute. Air flow will be measured using a magnehelic gage. The flow rate will be checked before, during, and after each sampling event. The PS-1 sampler will be calibrated before each sampling event using a manometer, calibrator, and the manufacturer's published calibration curve. The manufacturer's calibrator attaches directly to the top of the filter holder. The procedures followed during calibration will be as specified in the manufacturer's operating manual. A copy of the manual will be stored with the sampler.

As the PS-1 sampler is set up for sampling, a preweighed Teflon® filter and PUF plug cartridge will be loaded into the upper portion of the sampling module following the clean handling procedures outlined in EPA analytical method TO-4. When all the samplers to be used at a site have been deployed and a sampling event is imminent, the Teflon® filters and PUF plug cartridges for each sampler will be brought to the field and installed in each PS-1 sampler.

The air sample flow rate through the PS-1 sampler will be calibrated after the first few minutes of operation. The calibration will be conducted using the calibrator provided with the sampler in accordance with the manufacturer's operating manual. After calibration is completed, the serial number of the sampler, the start date and time for sampling, and all relevant calibration data will be promptly recorded in a field logbook.

After the sampling event, the end date and time will be recorded in the field logbook for each sampler. The Teflon® filter will then be removed from each sampler using stainless-steel tweezers. The Teflon® filters will then be placed in clean petri dishes, sealed with white plastic tape, and clearly labeled. The PUF plug cartridges will be similarly removed, placed in clean glass bottles (either amber or foil covered to exclude light) and clearly labeled.

In the laboratory, the Teflon® filters will be carefully weighed to measure particulate levels on the filter. PCBs and pesticides will be removed from the PUF plug cartridges using Soxhlet extraction in accordance with EPA analytical method TO-4. The extracts will be analyzed using GC with electron capture detection (ECD) following the procedures outlined in EPA analytical method TO-4.

2.4.2 Volatile Organic Compounds and Asbestos

Before being used to collect samples, the PAS-3000 sampler will be calibrated in the laboratory using a soap film flow meter following the manufacturer's operating manual. A copy of the manufacturer's calibration specifications and calibration results for each project will be maintained in a laboratory logbook.

After a sampling event is scheduled, the necessary PAS-3000 samplers will be deployed in the field. Each CMS cartridge will be transported to the field in a screwtop glass storage container. Asbestos filters will be transported to the field in sealed plastic bags. The clean handling procedures outlined in EPA analytical method TO-4 will be followed for all sampling equipment.

The CMS cartridges and asbestos filters will be installed in the PAS-3000 samplers just before the beginning of a sampling event. After each PAS-3000 sampler is turned on, the serial number of the sampler and the date and time will be recorded in the field logbook.

After the samples have been collected, the PAS-3000 samplers will be turned off and the end dates and times will be recorded in the field logbook. The CMS cartridges will then be removed from the samplers, recapped, and placed in screwtop glass storage containers for transport to the laboratory. The asbestos filters will be resealed in plastic bags for transport to the laboratory.

The CMS cartridges will be analyzed using the procedures outlined in EPA analytical method TO-2 for thermal desorption GC/ECD and flame ionization detectors (FID). The asbestos filters will be analyzed using phase contrast microscopy (PCM) in accordance with federal Occupational Safety and Health Administration standards for asbestos monitoring. The laboratory analyst will document compliance with these standards in the laboratory logbook.

If Tenax GC cartridges are used, they will be collected and analyzed following the same procedures used for the CMS cartridges. If samples are collected in SUMMA® canisters, the air sample will be withdrawn from the canister in the laboratory and will be analyzed directly, using GC/ECD and GC/FID in accordance with EPA analytical method TO-14. No filters or cartridges are used with Summa canisters.

2.5 QUALITY CONTROL PROCEDURES

For every 10 air quality samples collected using each type of sampler, an additional sample should be collected and submitted for analysis as a field blank. These field blank samples are used to verify the detection limits of the sampler and to check for the presence of cross contamination. Field blank sample results should be presented along with the results for actual air quality samples. In addition, 10 percent of all samples taken should be duplicate samples. The results of these samples are used to measure the precision of the sample analysis.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

CALIBRATION OF AIR SAMPLING PUMP

SOP NO. 064

REVISION NO. 0

Last Reviewed: November 1999

K Riesing

Quality Assurance Approved

May 24, 1993

Date

1.0 BACKGROUND

Several instruments are available to calibrate low air flow rate. The soap bubble meter method is one example. An air sampling pump and bubble meter calibrator are used to calibrate sample collecting devices including filters, impingers, sampling tubes, and color detector tubes. It is important to note that if a sampling pump uses a variable area flow meter (rotameter) for flow rate indication, the calibrated flow rate often must be adjusted for the actual air pressure and temperature during sampling. A formula for determining the corrected flow rate is provided.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for calibrating a rotameter sampler using an SKC® digital calibrator (calibrator).

1.2 SCOPE

This SOP provides instruction on calibration of a rotameter sampler by comparing a known airflow through the rotameter sampler and through the SKC® soap bubble meter calibrator.

1.3 DEFINITIONS

None

1.4 REFERENCES

SKC Inc. "Universal Flow Sample Pump Model 224-PCXR7 Operating Instructions." Form #3764-REV 706.

SKC Inc. "Electronic Calibrator Model 712 Operating Instructions." Form #3792-Rev 8 11.

1.5 REQUIREMENTS AND RESOURCES

To calibrate an air sampling pump the following equipment is needed:

- Air sampling pump
- SKC® digital calibrator (soap bubble meter)
- Soap solution
- Temperature and pressure gauge

2.0 PROCEDURES

The following procedures are used to calibrate an sampling pump with an SKC® digital calibrator:

1. The air sampling pump calibration should be checked at the beginning, middle, and end of the sampling event to determine the original loss in calibration.

2. Place the glass bubble meter in the digital calibrator (Figure 1). In general, if the flow rate is 2 liters/minute (L/min) or greater, slide the glass bubble meter to its lowest position on the stand. For flow rates of 500 milliliters (mL) or less, slide the glass bubble meter to its highest position on the stand. For intermediate flow rates, a bubble meter position between the extremes may be best.

3. Through the lower gas inlet tube, fill the liquid chamber with soap solution to a level just below the inner glass tubing.

4. Attach the flexible tubing to the upper gas inlet tube. Make this connection with the shortest tubing length possible, and avoid kinks and bends for the most accurate measurements.

5. Test the sampler battery pack for full charge by turning the sampler on using the ON/OFF switch (Figure 1). Press the START/HOLD key then the Flow and Battery Check key. Adjust the flow to 2 L/min using the flow adjustment control. The display should indicate "battery OK" in the upper left-hand corner.

6. While in the battery test mode, connect the flexible tubing to the filter housing intake. Set the sampler to the desired flow rate using the flow adjustment control.

7. Moisten the entire inner surface of the gas bubble meter with the soap solution. To do this, draw bubbles upward by squeezing the latex bulb until the bubbles travel the entire length of the bubble meter without breaking.

8. Press the ON/RESET button on the digital calibrator to turn on the instrument. Wait until a "0" is displayed, indicating the instrument is ready. There should be no bubbles in the area of the sensor block when the instrument is first turned on or when reset is pushed.

9. Squeeze the latex bulb gently to generate soap film bubbles. While the bubbles are being timed through the sensor block, the bulb should not be touched or erroneous flow rates may result. When bubbles pass through the lower sensor in the sensor block, the "TIMING IN PROGRESS" symbol (" + ") should be displayed.

10. For flow rates above 2 L/min the auto-bubbler clamp should be used. After the bubble meter has been moistened place the clamp on the large part of the latex bulb with the open end up. With the gas flowing, lightly tighten the clamp until the bubbles begin to form. Adjust the clamp so that the bubbles are going through the tube one at a time. When adjusted properly, hands off operation is possible and will continue for a period of time. When the bubbles stop forming, tighten the clamp.

11. After the bubble passes the upper sensor in the sensor block, the display will read out the gas flow rate. The gas flow rates measured by the digital calibrator should be within 0.7 L/min of the flow rate on the sampler.

12. Repeat the determination at least twice more and average the three results.

13. Measure the air temperature.

14. Record the following data on a calibration sheet:

    - Flow rate
    - Pressure of air sampled
    - Air temperature
    - Atmospheric pressure
    - Serial number of the pump
    - Pump number
    - Date and name of sampler

15. The expression for the corrected flow rate is:

$$Q_1 = Q_2(P_C T_S / P_S T_C)^{0.5}$$

where

Q_1 = Corrected flow rate (L/min)

Q_2 = Calibrator flow rate (L/min)

P_C = Atmospheric pressure (kiloPascals or other pressure units)

P_S = Pressure of air sampled (same units as P_C)

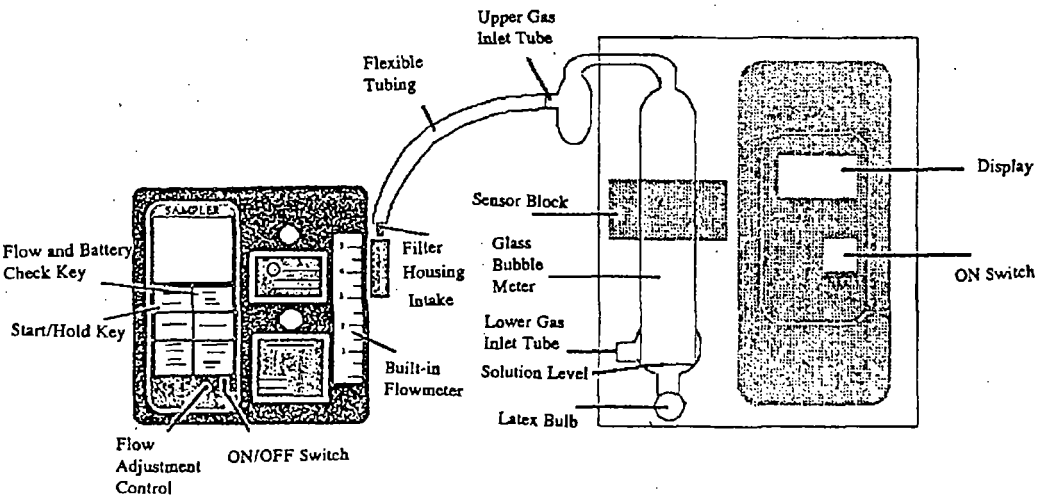
T_C = Temperature during calibration of sampling pump (Kelvin: •C + 273.16)

T_S = Temperature of air sampled (Kelvin: •C + 273.16)

The corrected flow rate is important to determine when sampling at high elevations or when temperatures are very low. The formula provided will help to determine the correct flow rate under such conditions.

FIGURE 1

AIR SAMPLING PUMP CALIBRATION APPARATUS



SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SOIL SAMPLING

SOP NO. 005

REVISION NO. 1

Last Reviewed: December 1999

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March 23, 1992

Date

1.0 BACKGROUND

Soil sampling is conducted for three main reasons. First, samples can be obtained for laboratory chemical analysis. Second, samples can be obtained for laboratory physical analysis. Third, samples can be obtained for visual classification and field screening. These three sampling objectives can be achieved separately or in combination with each other. Sampling locations are typically chosen to provide chemical, physical, or visual information in both the horizontal and vertical directions. A sampling and analysis plan is used to outline sampling methods and provide preliminary rationale for sampling locations. Sampling locations may be adjusted in the field based on the screening methods being used and the physical features of the area.

1.1 PURPOSE

Soil sampling is conducted to determine the chemical, physical, and visual characteristics of surface and subsurface soils.

1.2 SCOPE

This standard operating procedure (SOP) describes procedures for soil sampling in different areas using various implements. It includes procedures for test pit, surface soil, and subsurface soil sampling, and describes eight devices.

1.3 DEFINITIONS

Hand auger: Instrument attached to the bottom of a length of pipe that has a crossarm or "T" handle at the top. The auger can be closed-spiral or open-spiral.

Bucket auger: A type of auger that consists of a cylindrical bucket 10 to 72 inches in diameter with teeth arranged at the bottom.

Core sampler: Thin-wall cylindrical metal tube with diameter of 0.5 to 3 inches, a tapered nosepiece, a "T" handle to facilitate sampler deployment and retrieval, and a check valve (flutter valve) in the headpiece.

Spatulas or Spoons: Stainless steel instruments for collecting loose unconsolidated material.

Trier: Tube cut in half lengthwise with a sharpened tip that allows for collection of sticky solids or loosening of cohesive soils.

Trowel: Tool with a scooped blade 4 to 8 inches long and 2 to 3 inches wide with a handle.

Split-Spoon (or Split-Barrel) Sampler: Thick-walled steel tube that is split lengthwise. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to drill rods.

Thin-Wall Tube Sampler: Steel tube (1 to 3 millimeters thick) with tapered bottom edge for cutting. The upper end is fastened to a check valve that is attached to drill rods.

1.4 REFERENCES

Barth, D.S., and B.J. Mason. 1984. "Soil Sampling Quality Assurance Users Guide." EPA 600/4-84-043.

DeVara, E.R., B.P. Simmons, R.D. Stephens, and D.L. Storm. 1980. "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA 600/2-80-018. January.

Mason, B.J. 1983. "Preparation of Soil Sampling Protocol: Techniques and Strategies." EPA 600/4-83-020.

U.S. Environmental Protection Agency (EPA). 1987. "A Compendium of Superfund Field Operations Methods." OSWER Directive 9355.0-14 (EPA/540/P-87/001).

EPA. 1991. "Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells." March. EPA/600/4-89/034.

EPA. 1994. "Soil Sampling." Environmental Response Team SOP #2012 (Rev. #0.0, 11/16/94). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

Soil sampling requires that one or more of the following types of equipment be used:

Sampling Equipment	Other Required Equipment
Spoons and spatulas	Sample containers, labels, and chain-of-custody forms
Trowel	Logbook
Shovel or spade	Tape for measuring recovery
Trier	Soil classification information
Core sampler	Wax for sealing ends of thin-wall tube
Hand auger	Plastic sheeting
Bucket auger	Decontamination equipment
Split-spoon	Drilling equipment
Thin-wall tube	Backhoe
	Health and safety equipment

2.0 PROCEDURES

This SOP presents procedures for conducting test pit, surface soil, and subsurface soil sampling. The site sampling plan will specify which of the following procedures will be used.

Soil samples for chemical analysis should be collected in the following order: (1) volatile organics, (2) semivolatile organics, and (3) metals. Once the chemical samples have been containerized, samples for physical analyses can be containerized. Typical physical analyses conducted include (1) grain size distribution, (2) moisture content, (3) saturated permeability, (4) unsaturated permeability, and (5) Atterberg limits. Additionally, visual descriptions of samples, using the Unified Soil Classification System (USCS), should be recorded. Field tests such as head space analyses can also be conducted.

Soil samples for chemical analyses can be collected either as grab samples or composite samples. A grab sample is collected from a discrete location or depth. A composite sample consists of soil combined from more than one discrete location. Typically, composite samples consist of soil obtained from several locations and homogenized in a stainless steel or Teflon® pan or tray. Samples for volatile organic analysis (VOA) should not be composited.

2.1 TEST PIT SOIL SAMPLING

Test pit soil sampling is conducted when a complete soil profile is required or as a means of locating visually detectable contamination. This type of sampling provides a detailed description of the soil profile and allows for multiple samples to be collected from specific soil horizons. Prior to conducting any test pit or trench excavation with a backhoe, the sampling team should ensure that the sampling area is clear of utility lines, subsurface pipes, and poles.

A test pit or trench is excavated by incrementally removing soil material with a backhoe bucket. The excavated soil is placed on plastic sheeting well away from the edge of the test pit. A test pit should not be excavated to depths greater than 4 feet unless its walls are properly stabilized.

Personnel entering the test pit may be exposed to toxic or explosive gases and oxygen deficient environments. Air monitoring is required before entering the test pit and the use of appropriate respiratory gear and protective clothing is mandatory. At least two persons must be present at the test pit before sampling personnel enter the excavation and begin soil sampling.

Test pits are not practical for depths greater than 15 feet. If soil samples are required from depths greater than 15 feet, samples should be obtained using test borings instead of test pits. Test pits are also usually limited to a few feet below the water table. In some cases, a pumping system may be required to control the water level within the pits.

Access to open test pits should be restricted by use of flagging, tape, or fencing. If a fence is used, it should be erected at least 6 feet from the perimeter of the test pit. The test pit should be backfilled as soon as possible after sampling is completed.

Soil samples can be collected from the walls or bottom of a test pit using various equipment. A hand auger, bucket auger, or core sampler can be used to obtain samples from various depths. A trier, trowel, or spoons can be used to obtain samples from the walls or pit bottom surface.

2.2 SURFACE SOIL SAMPLING

The surface soil sampling equipment presented in this SOP is best suited for sampling to depths of 0 to 6 feet below ground surface (bgs). The sample depth, sample analyses, soil type, and soil moisture will also dictate the best suited sampling equipment. Prior to sample collection, the sampling locations should be cleared of any surface debris such as twigs, rocks, and litter. The following table presents various surface soil sampling equipment and their effective depth ranges, operating means (manual or power), and sample types collected (disturbed or undisturbed).

Sampling Equipment	Effective Depth Range (feet bgs)	Operating Means	Sample Type
Hand Auger	0 to 6	Manual	Disturbed
Bucket Auger	0 to 4	Power	Disturbed
Core Sampler	0 to 4	Manual or Power	Undisturbed
Shovel	0 to 6	Manual	Disturbed
Trier	0 to 1	Manual	Disturbed
Trowel	0 to 1	Manual	Disturbed
Spoon/Spatula	0 to 0.5	Manual	Disturbed

The procedures for using these various types of sampling equipment are discussed below.

2.2.1 Hand Auger

A hand auger equipped with extensions and a "T" handle is used to obtain samples from a depth of up to 6 feet. If necessary, a shovel may be used to excavate the topsoil to reach the desired subsoil level. If topsoil is removed, its thickness should be recorded. Samples obtained using a hand auger are disturbed in their collection; determining the exact depth at which samples are obtained is difficult.

The hand auger is screwed into the soil at an angle of 45 to 90 degrees from horizontal. When the entire auger blade has penetrated soil, the auger is removed from the soil by lifting it straight up without turning it, if possible. If the desired sampling depth has not been reached, the soil is removed from the auger and

deposited onto plastic sheeting. This procedure is repeated until the desired depth is reached and the soil sample is obtained. The auger is then removed from the boring and the soil sample is collected directly from the auger into an appropriate sample container.

2.2.2 Bucket Auger

A bucket auger, equipped similarly as the hand auger, is used to obtain disturbed samples from a depth of up to 4 feet. A bucket auger should be used when sampling stony or dense soil that prohibits the use of a hand-operated core or screw auger. A bucket auger with closed blades is used in soil that cannot generally be penetrated or retrieved by a core sampler.

The bucket auger is rotated while downward pressure is exerted until the bucket is full. The bucket is then removed from the boring, the collected soil is placed on plastic sheeting, and this procedure is repeated until the appropriate depth is reached and a sample is obtained. The bucket is then removed from the boring and the soil sample is transferred from the bucket to an appropriate sample container.

2.2.3 Core Sampler

A hand-operated core sampler (Figure 1), similarly equipped as the hand auger, is used to obtain samples from a depth of up to 4 feet in uncompacted soil. The core sampler is capable of retrieving undisturbed soil samples and is appropriate when low concentrations of metals or organics are of concern. The core sampler should be constructed of stainless steel. A polypropylene core sampler is generally not suitable for sampling dense soils or sampling at an appreciable depth.

The core sampler is pressed into the soil at an angle of 45 to 90 degrees from horizontal and is rotated when the desired depth is reached. The core is then removed, and the sample is placed into an appropriate sample container.

2.2.4 Shovel

A shovel may be used to obtain large quantities of soil that are not readily obtained with a trowel. A shovel is used when soil samples from a depth of up to 6 feet are to be collected by hand excavation; a tiling spade (sharpshooter) is recommended for excavation and sampling. A standard steel shovel may be used for excavation; either a stainless steel or polypropylene shovel may be used for sampling. Soil excavated from above the desired sampling depth should be stockpiled on plastic sheeting. Soil samples should be collected from the shovel and placed into the sample container using a stainless-steel scoop, plastic spoon, or other appropriate tool.

2.2.5 Trier

A trier (Figure 2) is used to sample soil from a depth of up to 1 foot. A trier should be made of stainless steel or polypropylene. A chrome-plated steel trier may be suitable when samples are to be analyzed for organics and heavy metal content is not a concern.

Samples are obtained by inserting the trier into soil at an angle of up to 45 degrees from horizontal. The trier is rotated to cut a core and is then pulled from the soil being sampled. The sample is then transferred to an appropriate sample container.

2.2.6 Trowel

A trowel is used to obtain surface soil samples that do not require excavation beyond a depth of 1 foot. A trowel may also be used to collect soil subsamples from profiles exposed in test pits. Use of a trowel is practical when sample volumes of approximately 1 pint (0.5 liter) or less are to be obtained. Excess soil should be placed on plastic sheeting until sampling is completed. A trowel should be made of stainless steel or galvanized steel. It can be purchased from a hardware or garden store. Soil samples to be analyzed for organics should be collected using a stainless steel trowel. Samples may be placed directly from the trowel into sample containers.

2.3 SUBSURFACE SOIL SAMPLING

Subsurface soil sampling, in conjunction with borehole drilling, is required for soil sampling from depths greater than approximately 6 feet. Subsurface soil sampling is frequently coupled with exploratory boreholes or monitoring well installation. Refer to SOP Nos. 045, 046, and 047 (borehole drilling SOPs) and SOP No. 020 (Monitoring Well Installation).

Subsurface soil sampling may be conducted using a drilling rig or power auger. Selection of sampling equipment depends upon geologic conditions and the scope of the sampling program. Two types of samplers used with machine-driven augers—the split-spoon sampler and the thin-wall tube sampler—are discussed below. All sampling tools should be cleaned before and after each use in accordance with SOP No. 002 (General Equipment Decontamination). Both the split-spoon sampler and the thin-wall tube sampler can be used to collect undisturbed samples from unconsolidated soils. The procedures for using the split-spoon and thin-wall tube samplers are presented below.

2.3.1 Split-Spoon Sampler

Split-spoon samplers are available in a variety of types and sizes. Site conditions and project needs such as large sample volume for multiple analyses determine the specific type of split-spoon sampler to be used. Figure 3 shows a generic split-spoon sampler.

The split-spoon sampler is advanced into the undisturbed soil beneath the bottom of the casing or borehole using a weighted hammer and a drill rod. The relationship between hammer weight, hammer drop, and number of blows required to advance the split-spoon sampler in 6-inch increments indicates the density or consistency of the subsurface soil. After the split-spoon sampler has been driven to its intended depth, it should be removed carefully to avoid loss of sample material. In noncohesive or saturated soil, a catcher or basket should be used to help retain the sample.

After the split-spoon sampler is removed from the casing, it is detached from the drill rod and opened. If VOA samples are to be collected, VOA vials should be filled with soil taken directly from the split-spoon sampler. Samples for other specific chemical analyses should be taken as soon as the VOA sample has

been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample (except for the top several inches of possibly disturbed material) is retained for analysis or disposal.

2.3.2 Thin-Wall Tube Sampler

A thin-wall tube sampler, sometimes called the Shelby tube (Figure 4), may be pressed or driven into soil inside a hollow-stem auger flight, wash bore casing, or uncased borehole. The tube sampler is pressed into the soil without rotation to the desired depth or until refusal. If the tube cannot be advanced by pushing, it may be necessary to drive it into the soil without rotation using a hammer and drill rod. The tube sampler is then rotated to collect the sample from the soil and removed from the borehole.

After removal of the tube sampler from the drilling equipment, the tube sampler should be inspected for adequate sample recovery. The sampling procedure should be repeated until an adequate soil core is obtained (if sample material can be retained by the tube sampler). The soil core obtained should be documented in the logbook. Any disturbed soil is removed from each end of the tube sampler. If chemical analysis is required, VOA samples must be collected immediately after the tube sampler is withdrawn. Before use, and during storage and transport, the tube sampler should be capped with a nonreactive material. For physical sampling parameters, the tube sampler should be sealed by pouring three 0.25-inch layers of sealing liquid (such as wax) in each end, allowing each layer to solidify before applying the next. The remaining space at each end of the tube is filled with Ottawa sand or other, similar sand, which is allowed to settle and compact. Plastic caps are then taped over the ends of the tube. The top and bottom of the tube sampler should be labeled and the tube sampler should be stored accordingly.

FIGURE 1

HAND-OPERATED CORE SAMPLER

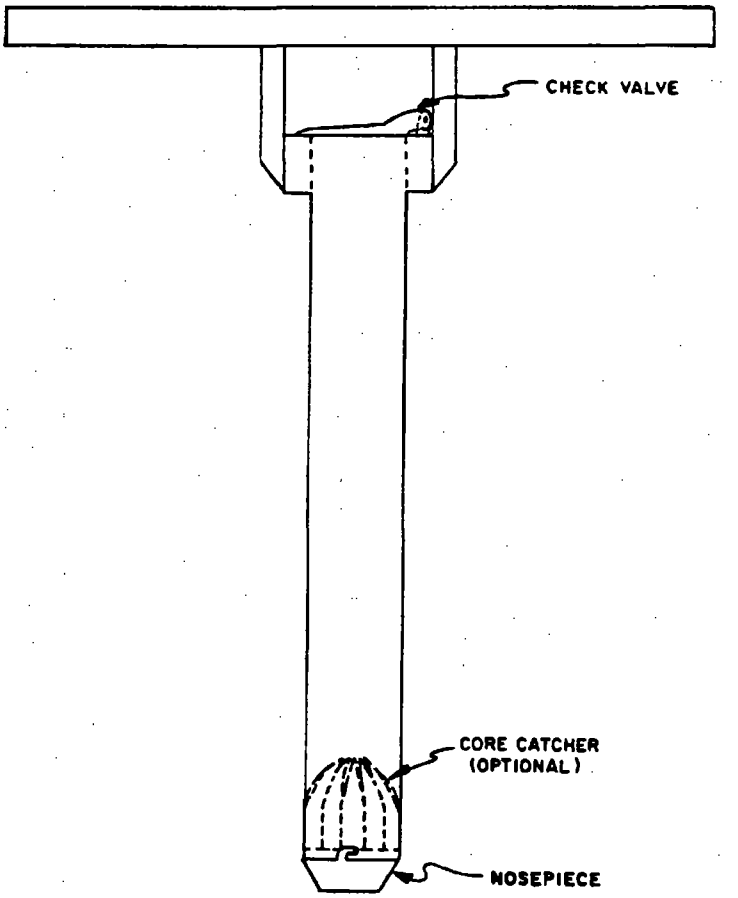


FIGURE 2

TRIER

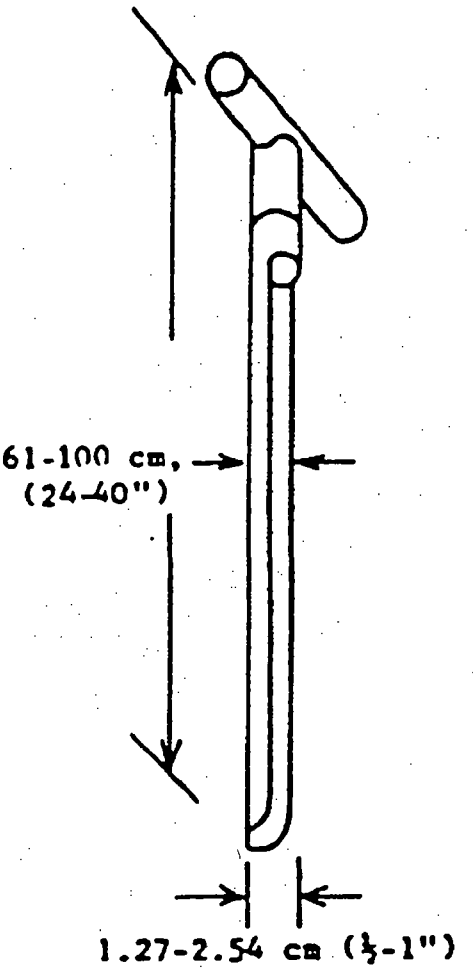


FIGURE 3

GENERIC SPLIT-SPOON SAMPLER

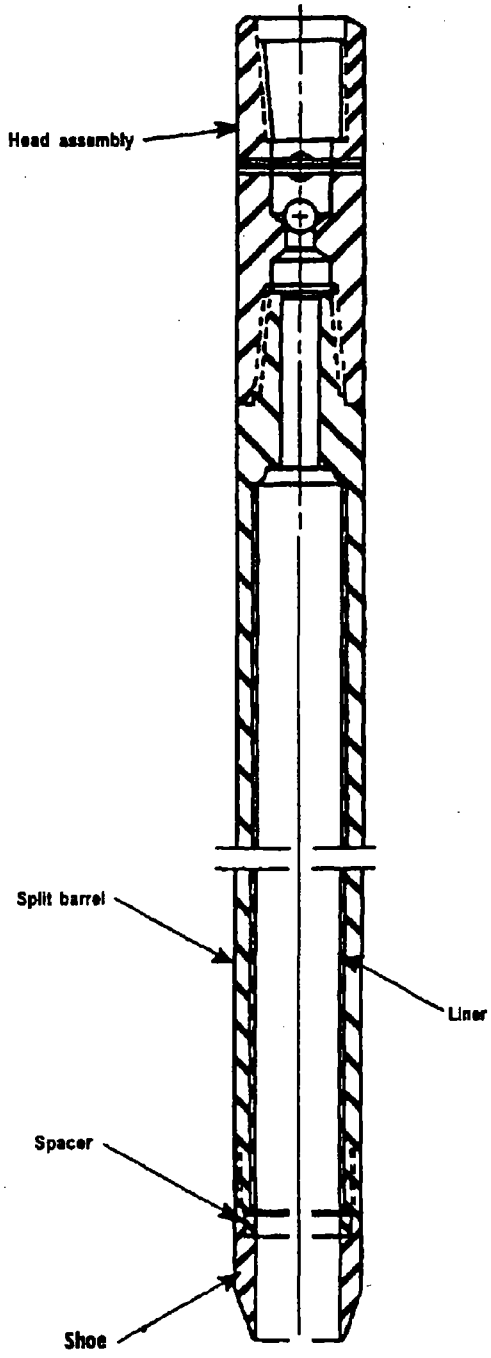
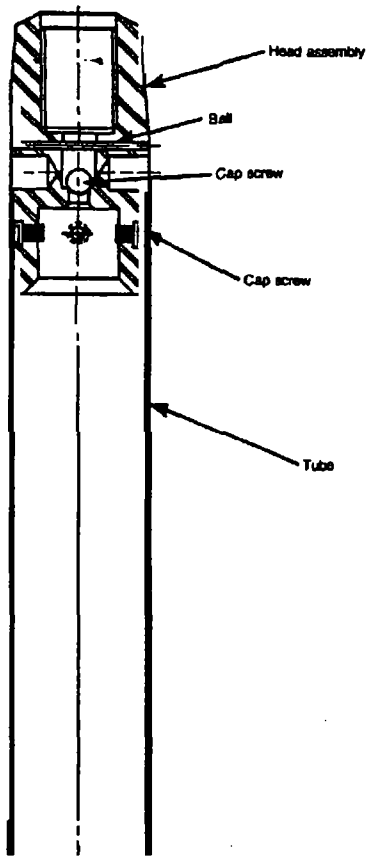


FIGURE 4

THIN-WALL TUBE SAMPLER



SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

USING THE GEOPROBE SYSTEM

SOP NO. 054

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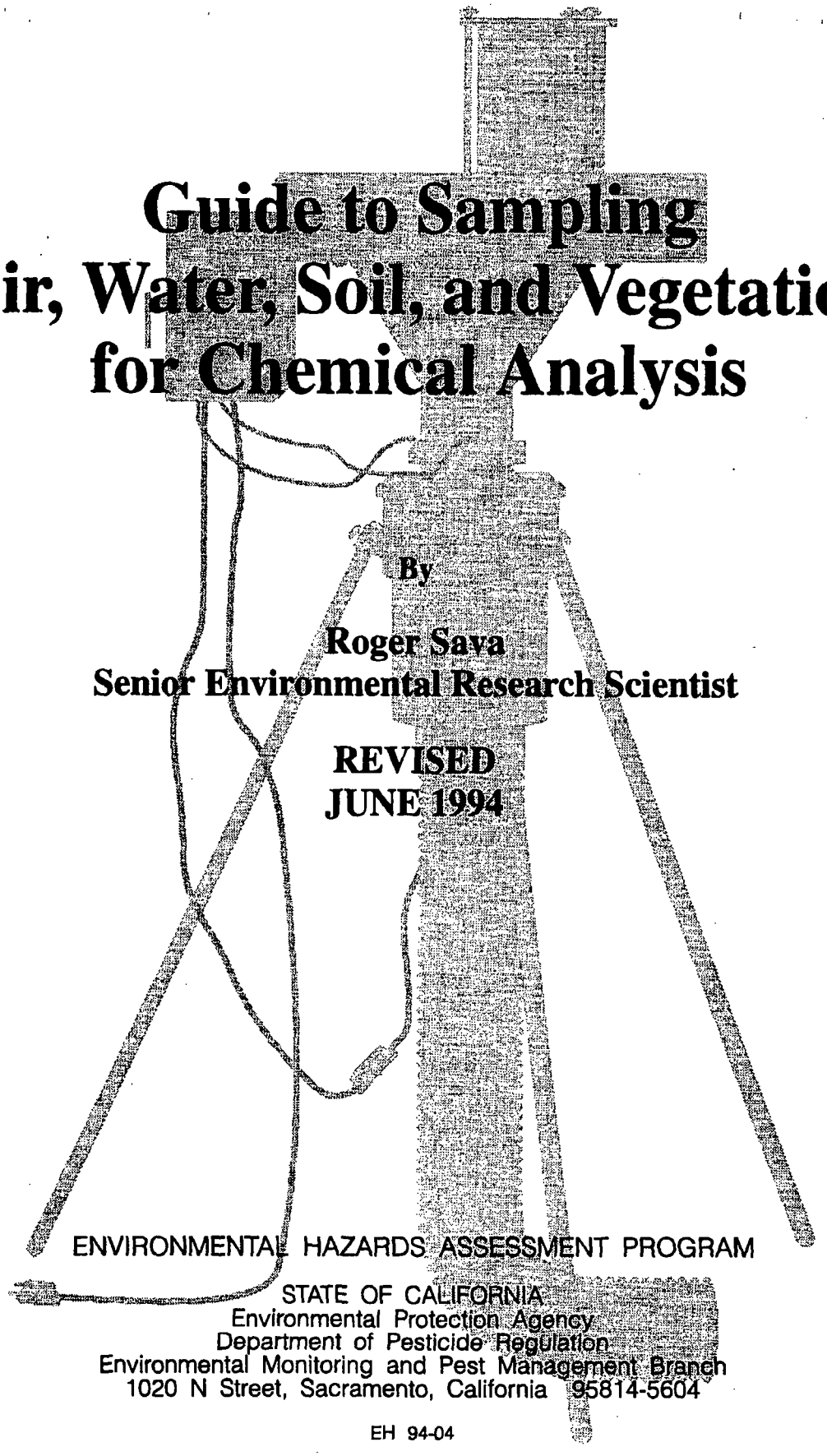
Date



Guide to Sampling Air, Water, Soil, and Vegetation for Chemical Analysis

By

Roger Sava
Senior Environmental Research Scientist

REVISED
JUNE 1994

ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM

STATE OF CALIFORNIA
Environmental Protection Agency
Department of Pesticide Regulation
Environmental Monitoring and Pest Management Branch
1020 N Street, Sacramento, California 95814-5604

EH 94-04

Acknowledgments

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Disclaimer

The mention of commercial products, their source or their use in connection with material reported herein is not to be construed as either an actual or implied endorsement of such product.

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LIST OF FIGURES

INTRODUCTION

This sampling guide was developed by the Environmental Hazards Assessment Program (EHAP), Department of Pesticide Regulation (DPR), California Environmental Protection Agency (Cal/EPA). It is intended to serve as an introductory guide to the collection of environmental samples for pesticide residue analysis. We suggest that this guide be supplemented with formal training courses in environmental monitoring. Other objectives, such as monitoring for compliance with California pesticide use regulations, may require sampling methods not included in this booklet.

The materials and techniques described in this booklet are given so that the user may be better equipped to collect high integrity environmental samples for chemical analysis of pesticide residue. Sampling methods should always have the overriding objective of obtaining samples of the highest possible integrity. A high integrity sample should provide, at the time of analysis, the best opportunity of determining the amount of chemical present in that medium as was present at the time the sample was obtained. Sampling should always be conducted with this objective in mind. When sampling for high levels of pesticides, follow label safety precautions.

Outlined in the guide are methods for collecting samples for pesticide residue analysis in air, surface water, ground water, soil, and vegetation. For each of these media, the sampling process is divided into six components: (1) an overview of the medium; (2) equipment and supplies; (3) site selection; (4) collecting the sample; (5) quality assurance/quality control; and (6) shipping and storing the sample. If technical assistance is needed or any questions arise, our staff is available at the following phone number:

(916) 324-4100

I. AIR SAMPLING

Overview

Air samples are collected by using any one of a number of commercially available sampling pumps. Our program makes use of three general categories of air samplers: High volume (Hi-Vols, Kurz Instruments), low volume (Lo-Vols, Anderson Samplers Inc.), and personal samplers (SKC West) (Figure 1). In general, Hi-Vol samplers have a high ratio of air flow to trapping medium and are used to measure low concentrations of pesticides (parts per billion (ppb) range or less) over long periods of time (1 to 24 hours or more). LoVol samplers have an intermediate ratio of air flow to trapping medium and may be used to measure higher pesticide concentrations, (ppb to parts per million (ppm) range) over shorter periods of time (from less than one hour to 12 hours or more). Low volume personal pumps have a low ratio of air flow to trapping medium and are used for air monitoring or measuring worker exposures at air flows of five liters per minute or less for pesticide concentrations in the high ppm to low ppb range, with run times from a few minutes to 8 hours or more. These are not hard, and fast rules, field conditions often dictate which sampler is best.

All air samplers draw air through a glass or stainless steel cylinder containing a sampling medium capable of trapping the chemical of interest. The various sampling media available consist of numerous sizes and types of sorbent resins, charcoal, and filters. These are used alone or in many combinations. Sampling cylinders for Lo-Vols and Hi-Vols can be prepared from component materials available from various vendors (Appendix D). All materials that will come into contact with samples are prepared by washing, double rinsing with deionized water, rinsing with a solvent (pesticide grade propyl or ethyl alcohol), and heat drying. A variety of pre-packed sorbent tubes, designed for use with the personal samplers, are available through SKC West (Appendix D).

Equipment and Supplies

You may find the following materials useful for air sampling:

- **EHAP Chain of Custody (COC) (Appendix A) or Request for Analysis Form 531-002 (Appendix B)** to document sampling history from sample generation to final analysis
- **Ball point pens** to fill out paperwork and record field notes.
- **Air Samplers: Lo-Vols or Hi-Vols** for sampling inside or outdoors, **Personal Samplers** for sampling worker exposure or when low volume flows are required (Appendix D).
- **Sampler calibration equipment** for adjusting and correcting flow rates.
- **Sample media** (resin jars and tubes) to collect air samples.
- **Timer** to turn samplers on/off at designated times.
- **Portable generator** to provide power for air samplers.
- **Extension cord** to connect electric source to samplers.
- **Duct tape** to hold extension cords in place.
- **Disposable gloves** to prevent sample contamination.

- **Silicon grease** to attach sample media tubes onto the samplers.
- **Plastic bags** and twist ties to package air samplers.
- **Ice chest to provide** storage and security of samples.
- **Dry ice** to keep air samples cold during shipment and storage.
- **Weather recording instruments** to document wind speed and direction.
- **Field note book** to document personnel, field locations, events, and any other pertinent information.
- **Label tape** to mark and identify sample container with the appropriate COC.

Site Selection

Air sampling indoors: Use Lo-Vol or Hi-Vol samplers depending on expected pesticide concentrations. Air samplers produce a moderate noise level during operation and in order to ensure that it will not later become a nuisance, let residents listen to a machine prior to actual sampling.

Hi-Vol samplers should be vented out of the dwelling to ensure that air will not be recycled through the machine causing erroneous results.

Avoid rooms with cigarette smoke or gas appliances; any gases or suspended smoke particles in the area may be trapped in the sampling medium and interfere with chemical analysis.

Air sampling outdoors: Hi-Vol or Lo-Vol air samplers may be used in outdoor situations.

The intake openings for the sampling tubes or jars should always be positioned to avoid exposure from engine exhausts, running motors, cigarette smoke, or any other non-target air contaminants.

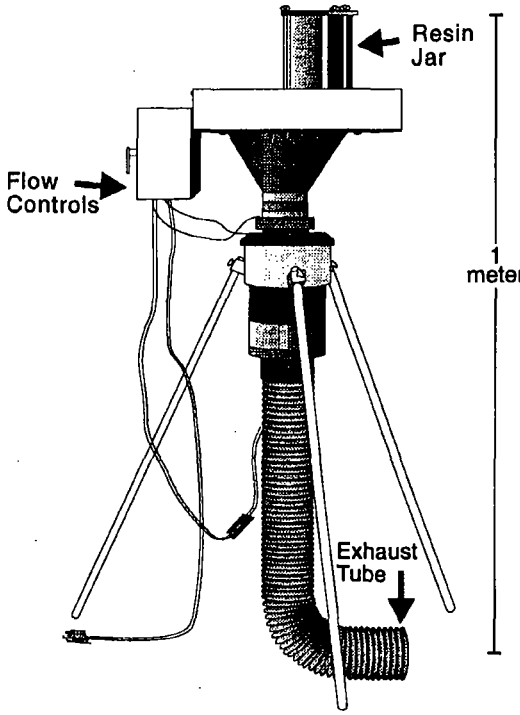
Protect samplers from rain and direct pesticide spray by using shelter hoods and by mounting sample tubes in a horizontal position.

Choose an area where the equipment will remain secure and not be subject to vandalism. The use of automatic timers to turn the samplers on and off may enable samplers to be used in some otherwise inaccessible locations. An example would be an area that is locked and only accessible during certain hours.

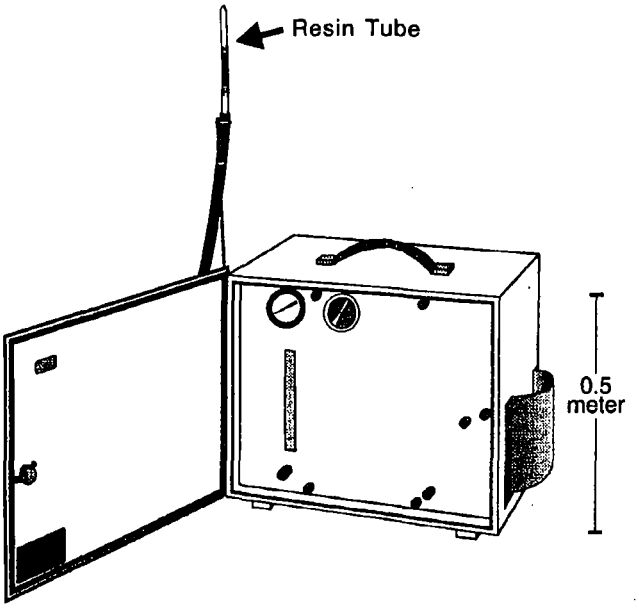
Collecting the Sample

A hands-on practice session may be necessary prior to operating air sampling equipment.

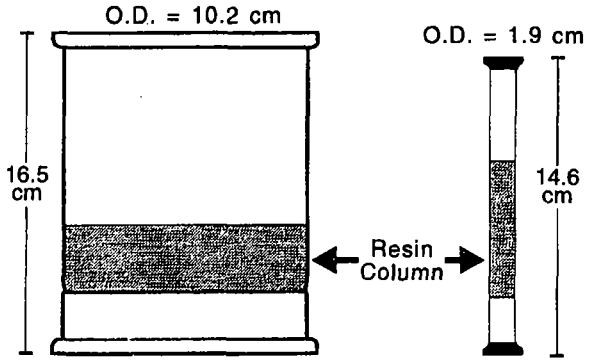
Determine the best trapping medium for the chemical of interest by consulting with the analytical lab, scientific literature, or sampling material guides available from vendors such as SKC West (Appendix D).



High Volume (HI-VOL) Air Sampler
Kurz Instruments

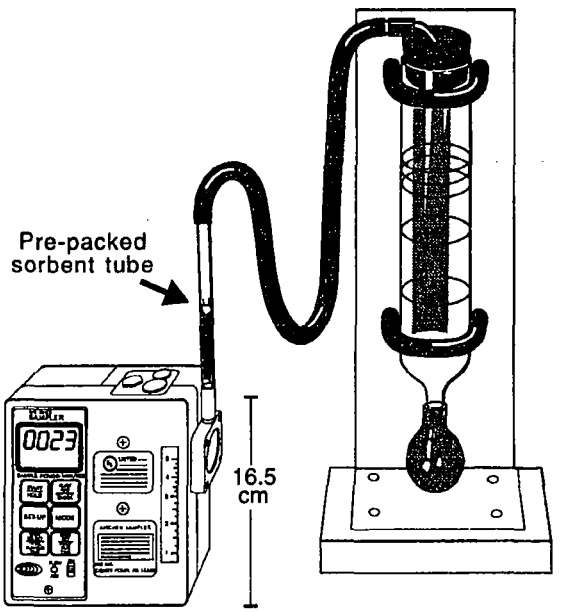


Low Volume (LO-VOL) Air Sampler
Anderson Samplers, Inc.



HI-VOL
Resin Jar
(125 ml Resin)

LO-VOL
Resin Tube
(15 ml Resin)



Low Flow Personal
air sampler
SKC Inc.

Bubble Calibrator
SKC Inc.

Figure 1. Types of Air Samplers

Use a new pair of disposable latex, plastic, or rubber gloves when handling sampling materials. Sample tubes and jars can be wrapped with aluminum foil to help prevent chemical breakdown by sunlight.

Be certain that the electrical power source is reliable; use portable generators when necessary and position them a sufficient distance away to avoid drawing exhaust fumes into sample containers.

When sampling air to determine the concentration of a chemical due to an application, it is best to determine background levels of that chemical immediately prior to the pesticide application. If possible, background air samples should run a length of time equal to the longest anticipated post application period sample.

Typical sample run times may vary from less than one hour to 24 hours or more; the distance from the pesticide source to the sampler site can vary but is generally within 100 meters, and the flow rate of a sampler can range from milliliters per minute to 1000 liters per minute or more. For a very general rule of thumb, expected concentrations in the parts per trillion (ppt) to low ppb range would indicate a choice of longer run times, shorter distances from pesticide source to sample sites, and higher flow rates. Expected concentrations in the high ppb to ppm range would indicate shorter run times, longer distances from pesticide source to sample sites, and lower flow rates. It is useful to document wind speed and direction during all sampling periods for outdoor air sampling, as the relationship between these parameters and sampler location will aid in interpreting pesticide concentration values.

Fill out a Chain of Custody (COC) form (Appendix A), or a Request for Analysis (Form 531-002) (Appendix B) for air samples. A COC is a legal form that is signed by all persons handling a sample and documents the custody of a sample from the time the sample container is prepared to the time the sample is analyzed. Make sure that all sample tubes are correctly marked and matched with the appropriate COC. Record other information on the COC including: the date and time sampler is started and stopped, persons collecting sample, flow rate of sampler, machine number, location of sampler, chemicals to be analyzed, and any other information that may affect the integrity of the sample.

Quality Assurance/Quality Control (QA/QC)

QA/QC samples are incorporated into air sampling investigations in order to enhance sample integrity, increase the confidence of analytical data, and to prevent false positives caused by contamination.

One type of sample that can be easily incorporated into an air sampling study is a "trip blank." A trip blank is a sample tube that is uncapped and recapped at the sample site, stored and transported with the other sample tubes, and submitted to the lab for analysis. The resulting expected "nondetected" (ND) analysis would increase the confidence that samples were not contaminated during preparation, shipping, storage or analysis.

In the process of extracting and analyzing the air samples, the lab should perform its own internal QC. When negotiating with the lab prior to submitting samples, request that they provide a copy of the internal QC performed with your samples and include this information in your report.

Shipping and Storage

Fit the open ends of the sample tubes with rubber stoppers or plastic caps and place in plastic bags. If resin jars are used, double wrap the jars in plastic bags and secure with rubber bands.

Immediately place samples in a container with dry ice. Place cardboard or paper on top of sample containers then place the dry ice on top to prevent breakage and to maximize cooling.

Ship and store air samples on dry ice (-70°C) until analysis. Less volatile compounds will store well at standard freezer temperatures of -10°C. Consult the analyzing chemist to determine the volatility or storage stability of the pesticide of interest.

It is preferable to store pesticide residue samples at -70°C. If dry ice is not available, use any form of refrigeration in the following order of desirability: 1) freezer, 2) refrigerator, 3) blue ice, 4) wet ice.

Always use an ice chest or container that has never been used to store concentrated pesticides or diluted formulations.

II. SURFACE WATER SAMPLING

Overview

Most surface water samples are collected by filling new, one-liter, narrow neck, amber glass bottles with Teflon®-lined caps. Bottles used previously are washed with detergent, double-rinsed with distilled water, rinsed with pesticide grade ethyl or propyl alcohol, and oven dried. If Teflon®-lined caps are not available, caps can be lined with aluminum foil to prevent contamination. Any type of one-liter glass containers with foil-lined caps, prepared as described, can be substituted as sample containers, and any of the sampling items listed below must also be prepared in this manner if they will come into contact with the sample. Some pesticides may bind to glass surfaces and must be collected in some other type of container. Consult with the analytical laboratory before choosing the type of container. We recommend using new containers whenever possible.

Equipment and Supplies

You may find the following materials helpful for surface water sampling:

- **EHAP Chain of Custody (Appendix A) or Request for Analysis Form 531-002 (Appendix B)** to document sample history from sample generation to final analysis.
- **Ball point pens** to fill out paperwork and record field notes.
- New one-liter narrow neck amber glass **sample bottles** with Teflon®-lined caps to contain water samples.
- A **Scoop** or a **"top off jar"** to completely fill sample bottle.
- **Hand pumps** can be useful for sampling water profiles and are available from scientific supply outlets.
- **pH meters** are needed if documenting the acidity of the sample is desired.
- **Preservatives** if required by the analyzing lab for the pesticide of interest.
- Equipment for measuring discharge and velocity of moving water bodies, (Appendix D).
- **Sharpie® pen** or other water-insoluble ink pen for labeling samples.
- **Aluminum foil may** be used to line bottle caps on sample jars that do not have Teflon® seals.
- **Disposable gloves** to help prevent sample contamination.
- **White tape** to label sample jars.
- **Boots or waders.**
- **Ice chest** to transport and store samples.
- **Wet ice** for storing and shipping samples.
- **Field notebook** to document personnel, field locations, events, and any other pertinent information.

Site selection

Use USGS 7-1/2 minute maps, which have excellent geographic detail, to identify tributaries and topographic features that may have some impact on the sample site.

When collecting from a stream, sample as close as is feasible to the suspected site of pesticide introduction (if known). Collect a composite sample (see page 10) from a transect of the river and submit one-liter sub-samples for analysis. Whenever possible, collect a stream sample prior to a convergent tributary to avoid dilution and channeling. Samples should also be collected from upstream tributaries or any other possible sources of pesticides flowing into the study area.

Collect a sample upstream from the suspected pesticide introduction in order to measure any background levels of the chemical of interest.

Determine the discharge and velocity of the stream and any tributaries. This information can be used to estimate the total mass of the contaminant and the rate of movement downstream. The equipment and methods needed to accurately measure stream discharge are described in Buchanan and Somers (1969). A variety of current velocity meters are available from various vendors. If you do not have equipment designed to measure stream discharge and velocity, you can approximate these measurements with the following formula using the float method described in Buchanan and Somers (1969):

Width of stream (feet) x average depth (feet) x speed of flow (feet per second) = flow rate in cubic feet per second

Collecting the Sample

Use shoulder-length, waterproof, disposable gloves if contact with water may pose a dermal exposure problem due to pesticides.

Prior to collecting the sample (while the bottle is still dry) mark each bottle using white labeling tape with a unique number in order to cross-reference the container with the appropriate COC or Form 531-002. Cover the label with clear tape and press firmly to assure the label will stay on the bottle.

Fill two bottles with water for each chemical or class of chemicals (e.g., organophosphorus). One of these bottles should be kept as a backup to be analyzed at a later time (e.g., primary bottle is lost or broken, or to confirm a positive analysis).

Avoid sampling from areas where water has been isolated from the main body of the stream, lake, or pond. Sample a stream transect while facing upstream; wade out as far as possible into ponds and avoid sampling the sediment that is disturbed by your movement.

After you have selected a sampling location, you can obtain a well-mixed sample by immersing the sample bottle, with the cap on, below the water surface. Remove the cap underwater and allow water to enter the bottle as you move the bottle vertically through the water profile. Avoid skimming the water surface unless that is your intent, (many substances with various degrees of solubility and specific gravity will float on a water surface and the sample may yield a concentration that is not representative of the entire water profile).

Fill bottle completely to eliminate all airspace (if an airspace is present in the bottle, the water-air interface may allow some chemical to vaporize prior to analysis). While the bottle is under water, replace the Teflon®- or foil-lined cap and bring the bottle out of the water.

If the body of water is too shallow to immerse a bottle, use a hand pump to draw water into the sample bottle. If a hand pump is not available, use a smaller, clean, glass container (i.e., a beaker or a jar) as a "scoop" and transfer water to the one-liter amber bottle.

Exercise caution so that the removed bottle cap does not come into contact with possible sources of contamination. A shirt pocket is a good spot to put one; the ground is not.

Several water samples collected throughout a pond or lake are preferable to a single grab sample collected at one location. If resources limit you to one sample, take several equal-size sub-samples from various areas throughout the body of water and pour them into a clean container. Thoroughly mix this **composite sample** and pour into the one-liter amber bottles, use a sample splitter whenever available to obtain a representative water split (Appendix D).

Record all information on a Chain of Custody (Appendix A) or on a Request for Analysis form 531-002 (Appendix B).

Quality Assurance/Quality Control (QA/QC)

QA/QC samples are incorporated into surface water investigations in order to enhance sample integrity, increase the confidence of analytical data, and to prevent reporting ("false") positives caused by contamination.

One type of QA/QC sample that can be easily incorporated into a surface water study is a "field blank." A field blank is a sample bottle that is prepared with the other bottles, packaged and transported to the sample site, filled with distilled or deionized water at the sample site, stored and transported with the other sample bottles, and submitted to the lab for analysis. The resulting expected "nondetected" (ND) analysis would increase the confidence that samples were not contaminated during preparation, field sampling, shipping, storage or analysis

QC "splits" are duplicate samples poured from a common container (one of the resulting samples from a composite split) at the sample site. Splits are handled the same as the primary samples, but are analyzed by a second laboratory and/or by a second analytical method.

A third type of QC sample is a "blind spike". Blind spikes are samples that are fortified with a known amount of the pesticide of interest and are generally prepared by the primary lab and stored with the field samples. When the field samples are delivered to

the lab, the QC spikes are included as unknowns, accompanied by a fictitious COC. The resulting analyses from split and spiked samples are used to confirm qualitative and quantitative laboratory results.

In the process of extracting and analyzing the field samples, the lab will also perform its own internal QC. When negotiating with the lab prior to submitting samples, request that they provide a copy of the internal QC performed with your samples and include this information in your report.

Shipping and Storage

Place samples immediately on wet ice (+4°C) for shipping and maintain at +4°C until analysis. Turbid or warm water samples that may have high bacteria populations can be salted with a pre-measured amount of table salt to prevent biodegradation. In some cases, other chemicals may be added or the pH may be adjusted to aid in preserving samples. If you have any question pertaining to, or if you are considering the use of, preservatives for a water sample, contact the laboratory that will do the analysis and discuss these options with a chemist. Addition of preservatives to a sample must be documented on the Chain of Custody or the Request for Analysis forms.

Always use an ice chest or container that has never been used to store concentrated pesticides or diluted formulations.

III. GROUND WATER SAMPLING

Overview

Ground water is usually sampled from existing water wells using new, one-liter, narrow-neck, glass amber bottles with Teflon®-lined caps. Many volatile compounds may require the use of volatile organic analysis (VOA) vials. Previously used bottles are washed with detergent, double-rinsed in distilled water, rinsed in pesticide grade ethyl or propyl alcohol, and oven dried. If Teflon®-lined caps are not available, caps can be lined with aluminum foil to prevent contamination. Any type of one-liter glass containers with foil-lined caps, prepared as described, can be substituted as sample containers, and any of the sampling items listed below must also be prepared in this manner if they will come into contact with the sample. Some pesticides may bind to glass surfaces and must be collected in some other type of container. Consult with the analytical laboratory before choosing the type of container. We recommend using new containers whenever possible.

Equipment and Supplies

You may find the following materials helpful for well water sampling:

- **EHAP Chain of Custody (Appendix A) or Request for Analysis Form 531-002 (Appendix B)** to document sample history from sample generation to final analysis.
- **Schrader® samplers** (Fig. 2 inset) made of stainless steel or Teflon® tubing, for sampling through a Schrader® valve.
- Extra **Schrader® valves and valve stems** to replace or repair defective valves.
- **Valve core remover** for removing Schrader® valves.
- **Teflon® tape** to reseal valves and fittings removed for sampling.
- **Adjustable wrench** for removing entire Schrader® valves, pipe plugs, and other fittings.
- Small **locking pliers** for securing Schrader® samplers to the Schrader® valve.
- Small flat **wood sticks** to override the electrical contact points in a pressure switch.
- **Plastic bags** to protect the electric points from contact with water.
- **Duct tape** to secure the plastic bags.
- **Garden hose** to direct water from hose bibs.
- **Five-gallon container** is useful when determining the delivery rate of a water well.
- **Deionized water** is used as the water source for QA/QC field blanks and for rinsing sampling equipment.
- **Alcohol** to rinse out Schrader® sampler tubes.
- **Department of Water Resources (DWR) Well Data Sheet (Form DWR 429)** to assist the DWR in its effort to locate and/or assign a permanent number to the well.
- **Ball point pens** to fill out paperwork and record field notes.
- New, one-liter, narrow-neck, amber glass, **sample bottles** with Teflon®-lined caps to contain water samples.
- **pH meters** are needed if documenting the acidity or basicity of the sample is desired.
- **Preservatives** if required by the analyzing lab for the pesticide of interest.
- **Aluminum foil** may be used to line bottle caps on sample jars that do not have Teflon® seals.

- **Disposable gloves** to help prevent sample contamination.
- **White tape** to label sample jars.
- **Ice chest** to transport and store samples.
- **Wet ice** for storing and shipping samples.
- **Field notebook** to document personnel, field locations, events, and any other pertinent information.
- **Sharpie® pen** or other insoluble marker for labeling sample bottles.
- **Polaroid® camera** to make a descriptive record of the well site.

Water Well Site Selection

Several criteria are listed here that may provide a sample that is representative of the supplying aquifer, and may minimize effects from water well construction. If you have a choice of wells to sample, choose one that meets as many of the following criteria as possible:

1) A driller's log for the well is available. Driller's logs contain valuable information about the construction and dimensions of the well at the time of construction including: depth to water-bearing strata; if any strata are sealed off; dimensions of the sanitary seal; placement of screens in the casing; depth of the bore hole; depth to standing water; and descriptions of construction materials.

2) Small domestic wells are preferable to large irrigation wells. Generally, domestic wells are drilled shallower, are sealed more carefully, and are less likely to contain contaminants often introduced by lubrication systems found on large turbine pumps.

3) A well casing constructed with steel is preferable to plastic or PVC (plastic can interfere with some pesticide analyses). However, more recent well construction is predominantly plastic.

4) The presence of a sampling port between the pump and the storage tank is preferable to a sampling port after the storage tank.

5) A well that is used regularly is preferred to one that is not in regular use.

6) The above-ground equipment and concrete pad should be in good condition. Check for cracks in the concrete pad, openings in the well head, water running into the well head, and storage of pesticides or other chemicals near the well head.

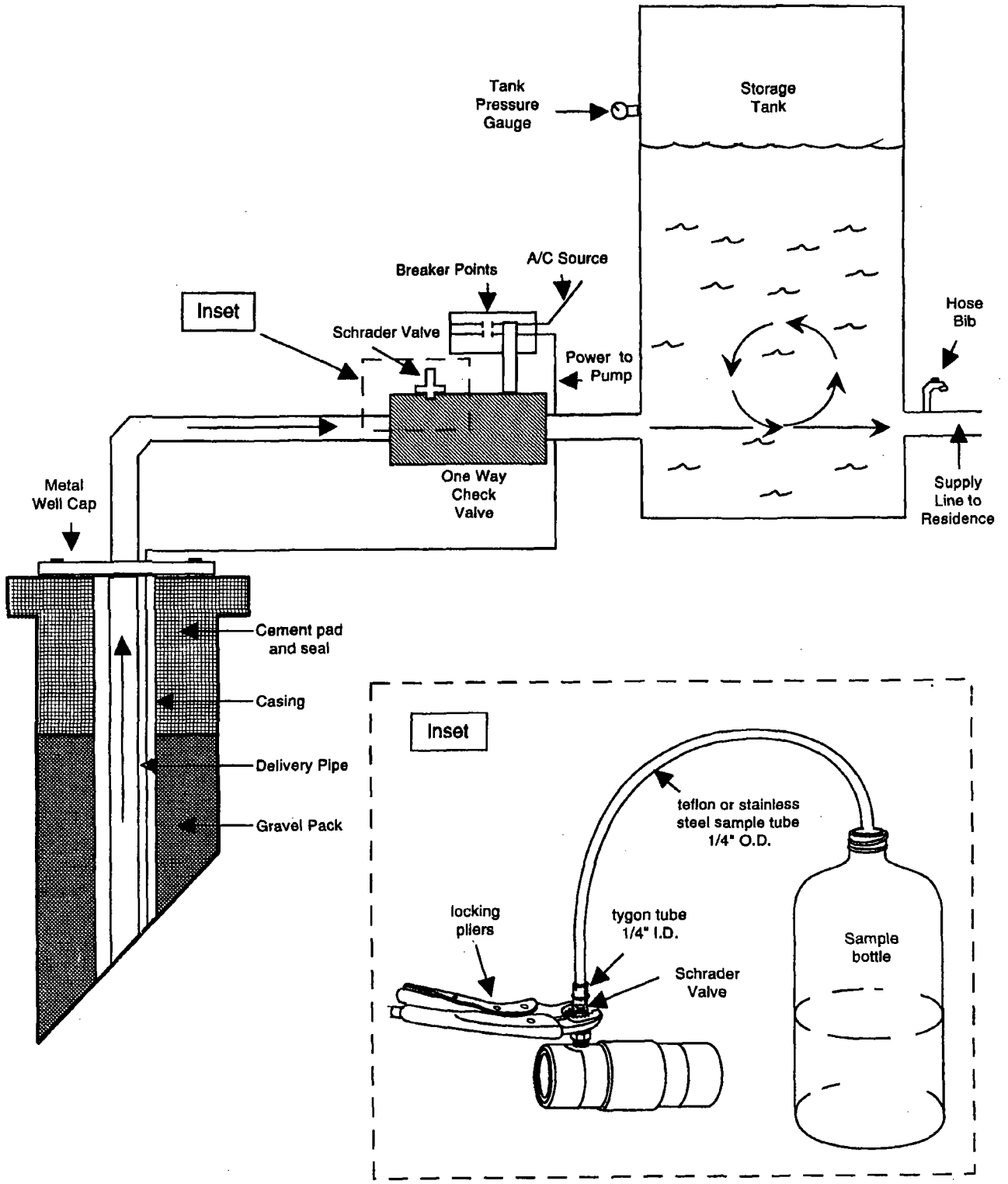


Figure 2. A typical domestic water well.

Obtaining the Sample

These instructions refer to a typical domestic water well shown in figure 2. Allow a well pump to run for a minimum of 10 minutes, or for the equivalent of three casing volumes, prior to taking the sample. When performing a 10-minute flush, open enough hose bibs around the house to ensure that the pressure gauge on the storage tank holds at a steady level below the shutoff pressure such that the pump output rate is equal to the system drain rate. If you are following a protocol that requires flushing three casing volumes, use a five-gallon container and calculate the amount of water delivered in one minute from all the open hose bibs while the system is holding at a steady pressure. Divide the equivalent of three casing volumes (in gallons) by the gallon per minute delivery rate to calculate the needed run time (to calculate casing volume in gallons, use the following formula: multiply πr^2 times the height of the standing water column, (π = 3.14, r = radius of casing, one ft^3 = 7.48 gallons)). These procedures will flush out the water which has stood in the casing and draw fresh water from the aquifer.

Every effort should be made to sample water prior to it entering the storage tank. The airspace and increased temperatures inside a storage tank could accelerate dissipation or degradation of many pesticides. If a pretank sample cannot be collected, take the sample from an outlet most closely plumbed to the wellhead. Sampling ports may include pipe plugs, Schrader® valves, faucets, or petcocks. When opening a system through one of these ports, you can interrupt the automatic off and on cycling by opening a circuit breaker, or by placing a thin stick between the breaker points. If you are familiar with typical domestic well equipment, you can follow the steps below to obtain a sample from a Schrader® valve. If not, we recommend that you call a qualified well repair person for assistance, or call one of our field staff for more detailed information.

Sampling from a Schrader® valve:

After running the pump for the desired time, turn faucets off and turn power to "pump off" (either by turning the main pump breaker switch to the off position or by interrupting the current through points).

If you must interrupt the current at the points, cover the point box with a plastic bag and secure with duct tape. Water in the points will short circuit the points and may damage the system.

Remove core from Schrader® valve and attach a **Schrader sampler**, (Tygon® with Teflon® or stainless steel tubing) and secure with locking pliers (Figure 2, inset).

Resume power to pump and fill sample bottles with water (the pump must be running in order to sample from the Schrader® valve). It may be necessary to leave some faucets on in order to keep the pump running. You may want to use a garden hose to direct water to an area where it will not pond up or create a problem for the homeowner.

After you have filled all of the sample bottles, turn off the power to the pump, replace the core in Schrader® valve and resume power to the pump.

Open faucets to reduce the pressure in the storage tank until the pump turns on, then close the faucet and allow the pump to run through one complete cycle to check that it is turning off and on properly and that there are no water leaks from the Schrader® valve core.

Record information on a well water Chain of Custody (Appendix C). If the well does not have a California Well Number, fill out a Well Summary Sheet (DWR form 429). Appendix E contains a blank DWR form 429. Make a copy of this form and carefully complete the following sections: Owner, Owner Address, Tenant, Tenant Address, Location-County, Township, Range, Section, Well Use. In the box marked "sketch," draw an accurate map of the well location, showing the distance in feet from the well to the center of the nearest two streets (reference and include a copy of the street map or if possible a USGS 7 1/2 minute topographical quadrangle map); north orientation; and any other wells on the property. Fill in any other information on the DWR form 429 that you observed or may have obtained from the well owner such as the casing material, pump type, year drilled or re-drilled, etc. Submit it to the nearest DWR district office. They will assign a well number and send it back to you. For more details on filling out DWR form 429, or for more information on numbering water wells in California, see Appendix E.

Quality Assurance/Quality Control (QA/QC)

QA/QC field samples are incorporated into ground water investigations in order to enhance sample integrity, increase the confidence of analytical data, and to prevent reporting positives caused by contamination.

One type of QA/QC sample that should be incorporated into ground water studies is a "field blank". A field blank is a sample bottle that is prepared with the other bottles, packaged and transported to the sample site, filled with distilled or deionized water at the well water sampling site, stored and transported with the other sample bottles, and submitted to the lab for analysis. The resulting expected "nondetected" (ND) analysis would increase the confidence that samples were not contaminated during preparation, field sampling, handling, shipping, storage, or analysis.

QC "splits" are duplicate samples poured from a common container at the sample site. Splits are handled the same as the primary samples, but are analyzed by a second laboratory and/or by a second analytical method.

"Blind spikes" are samples that are fortified with a known amount of the pesticide of interest and are generally prepared by the primary lab and stored with the field samples. When the field samples are delivered to the lab, the QC spikes are also included as unknowns, accompanied by a fictitious COC. The resulting analyses from split and spiked samples are used to confirm qualitative and quantitative laboratory results.

In the process of performing the analyses on the field samples, the lab will also perform its own internal QC. When negotiating with the lab prior to submitting samples, request that they provide a copy of the internal QC performed with your samples and include this information in your report.

Shipping and Storage

Immediately after filling containers, refrigerate well water samples for shipping and storage until analysis. Typical ice chest temperature of +4°C is preferable for storing samples in one-liter, amber glass bottles. However, some pesticide compounds may require freezing at -10°C or subfreezing temperatures to -70°C (dry ice), in these cases, a polycarbonate, polypropylene, or polyethylene bottle may be appropriate. In some situations, well water samples can be salted with a pre-measured amount of table salt to prevent biodegradation. In some cases, other chemicals can be added or the pH can be adjusted to aid in preserving samples. If you have questions pertaining to preservatives, storage temperatures, and sample container type for ground water samples; or if you need information regarding storage stability of the chemical of interest, contact the laboratory that will do the analysis. Remember to document addition of preservatives on the Chain of Custody or Request for Analysis form.

Always use an ice chest or bottle that has never been used to store concentrated pesticides or diluted formulations.

IV. SOIL SAMPLING

Overview

All soil and sediment samples are collected in one-quart, glass Mason jars. New jars need not be washed. Previously used jars are washed with detergent, double-rinsed in distilled water, rinsed in pesticide-grade ethyl or propyl alcohol, and oven dried. Mason jar lids should be lined with aluminum foil. All tools that come in contact with soil samples should be washed with detergent, rinsed in distilled water, and rinsed with alcohol prior to each sampling. Stainless steel is a good choice for shovels, buckets, and other sampling equipment.

Equipment and Supplies

You may find the following materials useful for soil sampling:

- **EHAP Chain of Custody (Appendix A) or Request for Analysis Form 531-002 (Appendix B)** to document sample history from sample generation to final analysis.
- **Soil augers** (Fig. 3), soil sampling tubes, slide hammer, and extruder or Acker® soil sampler, or other device to obtain soil cores.
- **PVC pipe** 4 inch inside diameter (ID) x 12 inches long to line the bore hole when using a bucket auger.
- **Rubber mallet** and 12 inch 2 in. x 4 in. wood blocks are used for inserting the PVC pipe.
- **Shovels** for taking soil samples may come in handy when all else fails.
- **Mixing containers** such as stainless steel buckets or large bags are useful when mixing composite samples.
- **Sample jars** for collecting, storing, and shipping samples.
- **Aluminum foil** to line sample jar lids.
- **White Labeling tape** to mark sample jars.
- **Cellophane tape** to seal over labeling tapes.
- **Ice chests**.
- **Dry ice** for storing and shipping soil samples.
- **Detergent** for cleaning equipment.
- **Cleaning brushes** to remove soil from sample equipment between samples.
- **Five-gallon buckets** or other suitable containers to hold wash and rinse solutions.
- **Alcohol** to decontaminate sample equipment after washing.
- **Distilled water** in sufficient amounts to rinse all sample equipment prior to collecting each new sample.
- **Wash bottles** for dispensing alcohol and water.
- **Paper towels** to aid in drying sampling equipment.
- **Disposable gloves** to prevent contamination and exposure to chemicals.
- **Boots**, in case the going gets muddy.
- **Sharpie® pen**, permanent marker, to label sample jars.
- **Field notebook** to document personnel, field locations, events, application history, and any other pertinent information.

Site Selection

If you have a choice, always try to randomize the selection of sample sites within a field. Keep in mind that some fields may have pesticide applications that, by intent, are not uniformly applied; in these cases, some areas in the field will have higher concentrations of chemicals.

Obtaining the Sample

Surface soil sampling:

Randomize soil sampling sites throughout a field. Take a composite soil sample: collect a pint of soil from 5 to 10 sites throughout the field, combine these sub-samples in a large bucket or plastic bag. Mix sample thoroughly, fill the one-quart sample jar, and discard the remaining soil.

Always wash sampling equipment before collecting each new sample that is not part of a composite sample. Remove all soil by washing with detergent and water, rinsing with distilled water, then rinsing with ethyl alcohol if available. Isopropyl alcohol can be used as a substitute. It is not necessary to wash sampling equipment while collecting sub-samples for a composite sample.

A soil sample marked "surface soil down to a maximum of five centimeters (cm.)" means that the sample contains soil taken from zero (soil surface) to five cm. deep. This sample can be collected with a hand trowel, shovel, pick, etc. A soil sample marked "zero to five cm. core" indicates that the sample was collected using a coring device which is driven into the ground and contains an equal amount of soil from the one, two, three, through five cm. depth. Indicate on the Chain of Custody which method was used to collect the soil sample.

Sampling soil to shallow depths:

Our program utilizes hand operated soil augers (also referred to as bucket augers) to sample soil down to a depth of 3 meters (Fig. 3). For sampling depths of one meter or less a variety of commercially available soil tubes can be used. Before digging, provide a detailed site map and check with local municipalities if the possibility of underground lines exist.

To sample the top 15 cm. (6 inches) of soil with a bucket auger:
Using a rubber mallet, drive a cylindrical PVC plastic sleeve into the soil to a depth of approximately 15 cm. (This first step is only necessary when sampling loose sandy soils, or when surface concentrations of suspected contaminants are expected to be high.)

The first 15 cm. sample is obtained by screwing the auger through the sleeve to the desired depth, then retrieving the auger and shaking the entire sample into a plastic bag. The sample can then be mixed in the bag and poured into a glass jar. If a composite sample is desired, all sub-samples from ground level to 15 cm. can be mixed in a bag and the resulting composite sample poured into a glass jar. Discard excess soil.

To sample deeper increments, manually remove excess soil from inside the sleeve to the 15 cm. depth wearing a clean plastic glove. The auger is then cleaned in soapy water, triple rinsed in deionized or distilled water, then rinsed with alcohol.

Subsequent samples are taken through the sleeve generally in 15 cm. increments; soil is not manually removed from the borehole for these subsequent samples.

Most of the loose soil that may have dropped into the borehole, and is now in the top end of the auger, can be removed from the filled bucket auger by striking the bucket with a rubber mallet while holding the auger parallel to the ground.

The procedures for mixing sub-samples in a plastic bag and pouring samples into jars is then repeated. Remember to line lids with foil. If the field to be sampled is shaped with furrows and beds, keep in mind that chemicals may have been applied in narrow bands. Thus, you may need to use a shovel to remove sections of soil perpendicular to the direction of furrows to ensure that the sample submitted will be representative of the field (Fig. 4). Sample soil to a depth of 5 to 10 cm. below the label application depth for the suspected pesticide. You may get inaccurate results if you use soil augers or soil coring tubes to sample a field that has been banded or spot treated.

Quality Assurance/Quality Control (QA/QC)

QA/QC field samples are incorporated into soil sampling investigations in order to enhance sample integrity, increase the confidence of analytical data, and to prevent reporting positives caused by contamination.

A "rinse blank" is a water sample collected by capturing distilled or deionized water that is poured over all the components of the soil sampling equipment. The rinse sample is collected after the equipment is cleaned according to directions in **"Obtaining the sample,"** paragraph 2, and prior to using the equipment to collect a soil sample. The rinse blank is then stored and transported with the other sample bottles and submitted to the lab for analysis. A "nondetected" (ND) analysis of a rinse blank would indicate that the cleaning procedures were adequate for preventing cross contamination from the sampling equipment.

Another type of QA/QC sample that can be incorporated into a soil sampling study is a "split." A pre-determined number of field samples are submitted to the primary lab with instructions to thoroughly mix the sample, split into two sub-samples, and analyze one sample. The other half of the split is then analyzed by a second lab. The resulting two analyses can then be compared.

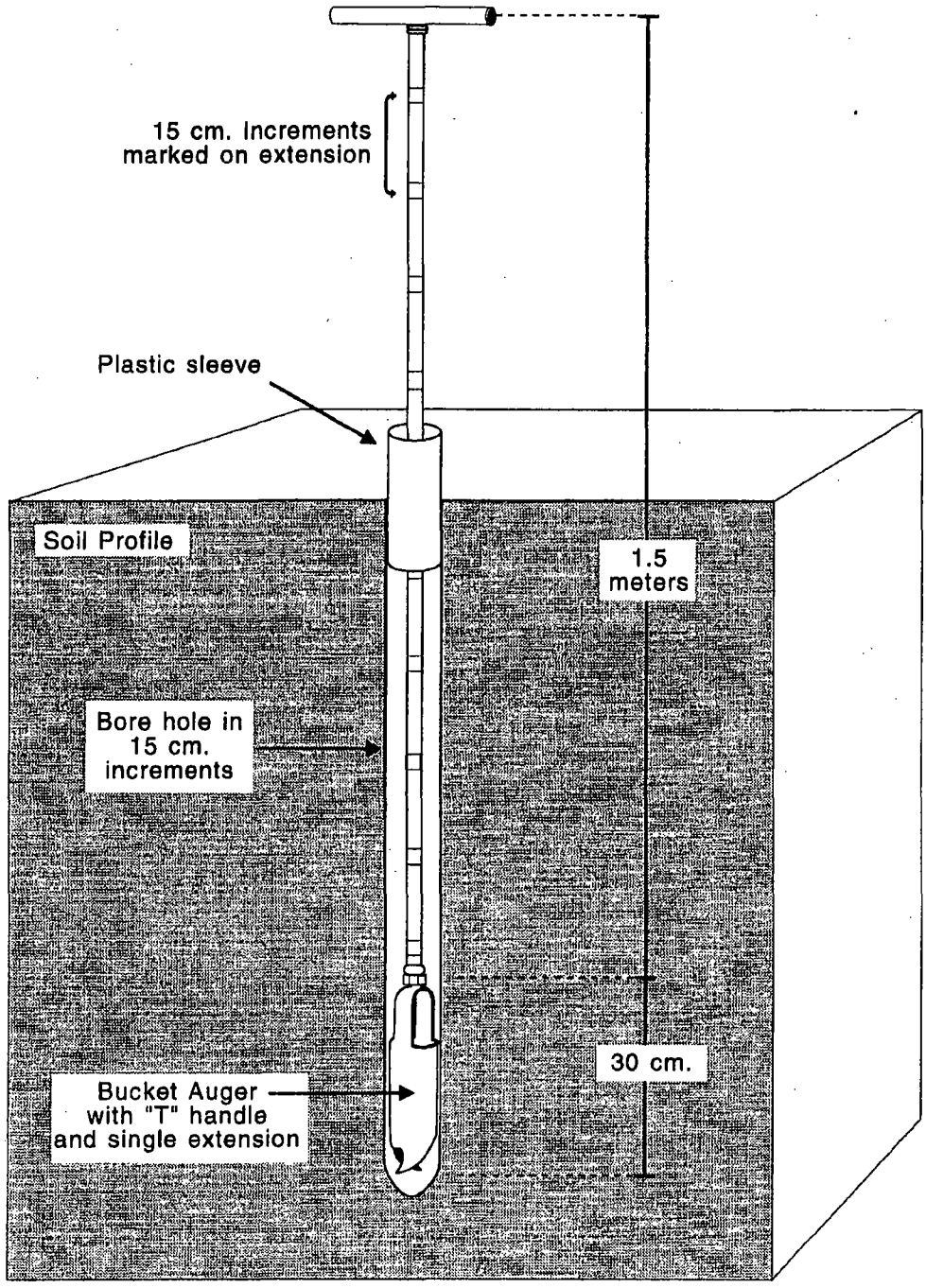
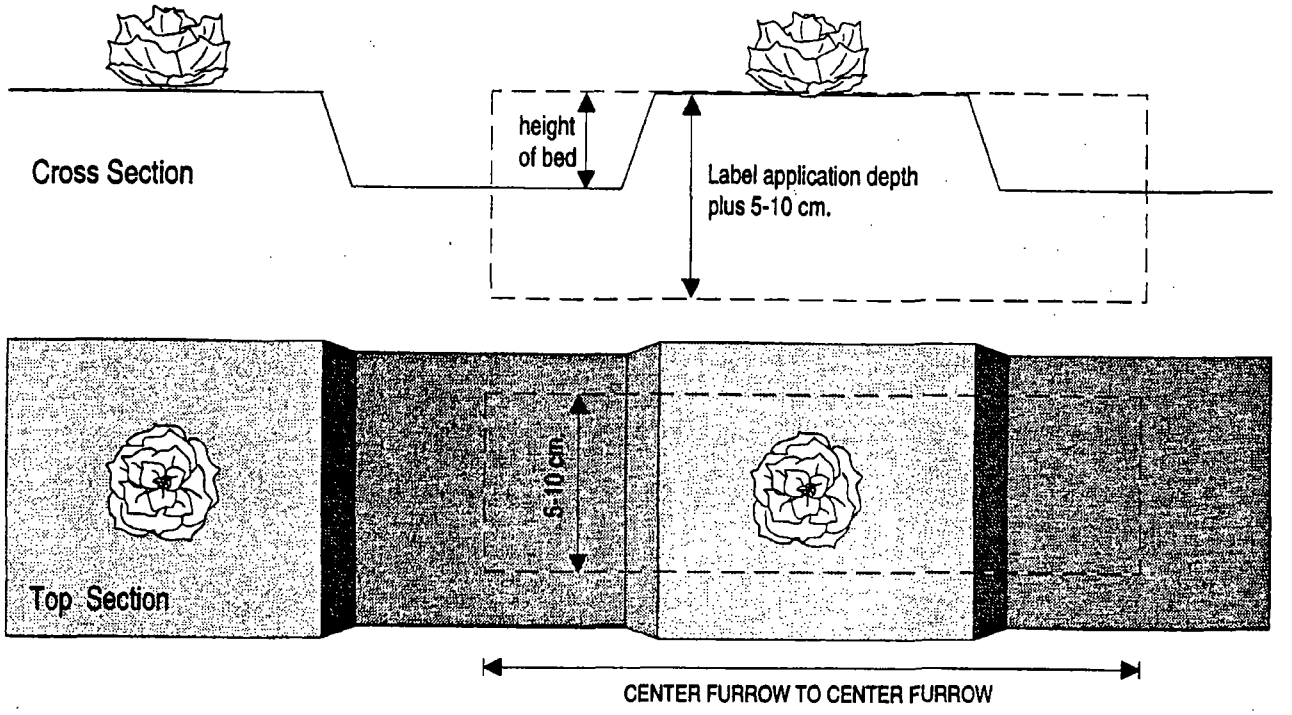


Figure 3. Sampling a soil column with a Bucket Auger.

A. SINGLE ROW BEDS:

COMBINE SOIL, FROM THE IMAGINARY AREA OUTLINED IN FIGURE A OR B, IN A CONTAINER AND FILL A 1 QUART JAR.



B. DOUBLE ROW BEDS

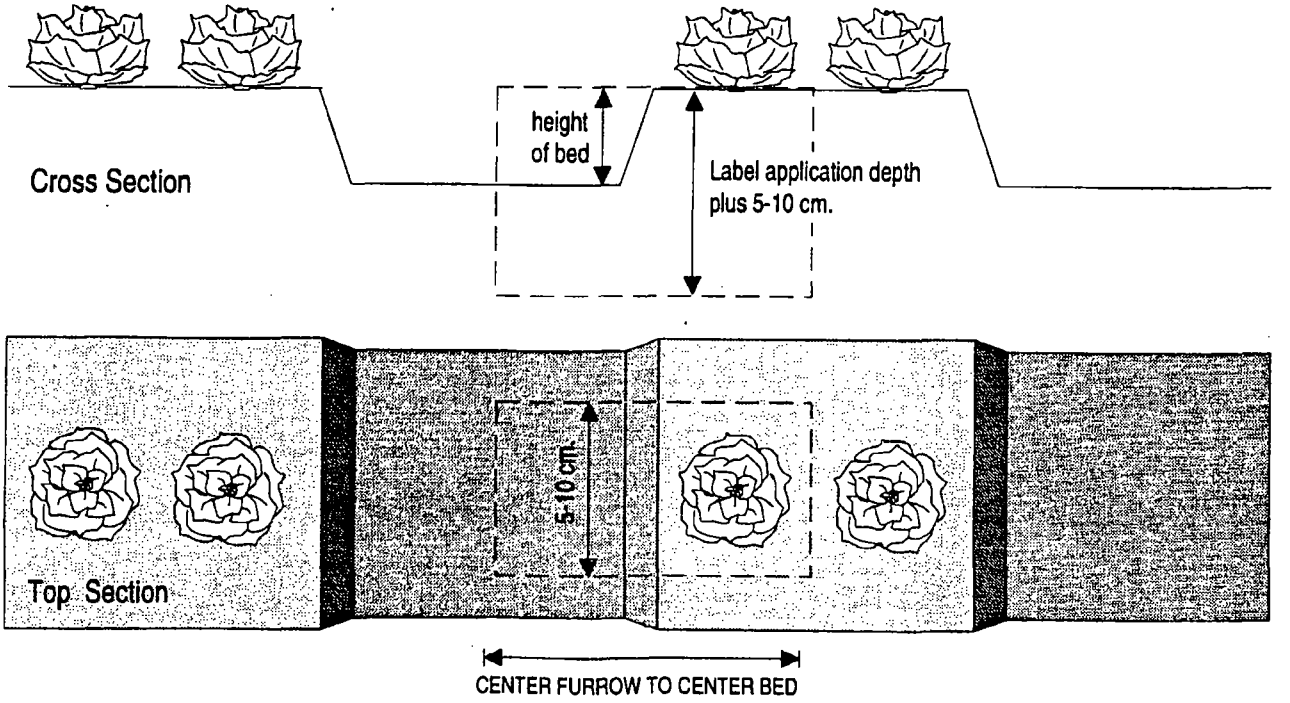


Figure 4. Soil Sampling in Furrowed Fields

A "blind spike" is a soil sample that is fortified with a known amount of the pesticide of interest and mixed thoroughly on a roller. Matrix spikes are submitted as unknown to the lab accompanied by a fictitious COC. The resulting analyses from splits and matrix spikes are used to confirm qualitative and quantitative laboratory results.
In the process of performing the analyses on the field samples, the lab should also perform its own internal QC. When negotiating with the lab prior to submitting samples, request that they provide a copy of the internal QC performed with your samples and include this information in your report.

Shipping and Storage
Ship soil samples at -70°C (dry ice). Store soil samples in a freezer. Ship and store rinse blanks at +4°C (wet ice or refrigeration).

V. VEGETATION/FRUIT SAMPLING

Overview

Vegetation may be sampled to determine dislodgeable residues and/or total residue. Dislodgeable residues are determined by analyzing residues from the surface of the foliage. The results are reported as a weight-to-surface area ratio. Total residue is determined by extracting and analyzing surface and internal residues from the vegetation sample, and the results are reported as a weight-to-weight ratio. When sampling for total residue, collect leaves in one-quart Mason jars or, for smaller samples, in one-pint jars. If you are using a leaf punch designed for collecting dislodgeable residue samples, the leaves are collected in the glass container screwed on to the punch. Cap off the container after the sample is collected and screw a new jar on to the leaf punch to collect the next sample.

Jars that are re-used should be washed with detergent, double rinsed in distilled water, rinsed in pesticide-grade ethyl alcohol, and oven-dried. New jars do not need to be cleaned. Caps should be lined with aluminum foil. All tools that come in contact with vegetation should be washed with detergent, rinsed in distilled water, and rinsed with ethyl alcohol prior to collecting each sample. We recommend using new containers for residue sampling.

Equipment and Supplies

Materials that you may find useful for vegetation/fruit sampling:

- **Request for Analysis Form 531-002 or EHAP Chain of Custody** to document sample history from sample generation to final analysis.
- **Glass jars** or **plastic bags** to contain samples during shipment and storage.
- **Disposable gloves** to prevent contamination of samples and to prevent chemical exposure.
- **Scissors** for removing vegetation from plants.
- **Leaf punch** for collecting dislodgeable residue samples.
- **Ladder** for sampling taller trees.
- **Pole picker** for reaching fruit high in trees.
- **Aluminum foil** to seal sample jar lids.
- **Detergent** for washing sampling equipment.
- **Alcohol** and **distilled water** to rinse sampling equipment.
- **Wash bottles** for dispensing alcohol and water.
- **Labeling tape** to mark sample bottles.
- **Clear cellophane tape** to seal labeling tape.
- **Ice chest** and **dry or wet ice** for storage and shipment of samples.

Site Selection

Sampling leaves from trees and shrubs:

Choose a plant that has enough foliage to provide sufficient material for the duration of the monitoring period.

To estimate the average concentration of a chemical over the entire plant, your sample should include foliage from all locations on the plant including the top, bottom, inside, and outside of all the sides.

To determine the direction of possible pesticide drift, take separate samples from the north, south, east, and west sides of the plant.

Sampling a row or field crop:
Randomly collect leaves from all sides of several plants, from different areas of a field and do not include leaves that have contacted the soil.

When sampling vegetation for a period of days (i.e., if the purpose of the investigation is to determine the dissipation rate of a pesticide over time), keep in mind that new growth after the chemical application may affect the results of the analysis.

Obtaining the Sample
Leaf samples:
Handling - Use a clean pair of disposable gloves when handling vegetation samples to prevent exposure to chemicals and to prevent cross-contamination of other samples. Handle leaves as little as possible. Always use clean scissors and, when possible, cut leaves off directly into the sample container. Leaf punches can also be used to obtain samples for dislodgeable residue analysis.

Sample Size - A reasonable number of leaves for a single sample is 30-40 small leaves, 20-25 medium leaves, and 15-20 large leaves. If a leaf punch is used, take 40-60 discs per sample and record the exact number on the COC.

Containers - Polyethylene bags are convenient containers for leaf samples but may not be suitable for all pesticides. Whenever possible, glass containers with foil-lined lids are recommended and, if requesting dislodgeable analyses, glass containers are required (a container rinse is part of the extraction process).

Fruit Samples:
Handling - Use a clean pair of disposable gloves when handling fruit samples.

Sample Size - One pound is a reasonable sample size.

Methods - The sample will be more representative if it contains fruit collected from several areas of the field. When sampling plant parts that grow above ground, avoid parts which come in contact with soil. When sampling plant parts that grow underground, such as sugar beets or potatoes, extra precautions must be taken to avoid piercing the underground stem or root with sampling tools because the surrounding soil may contain pesticides in concentrations high enough to contaminate the sample. Wash samples which grow underground before placing them in the sample container (to avoid contamination by pesticide-laden soil), but do not wash fruits which grow above ground.

Containers - Fruits (including nuts) should be packaged in glass containers whenever possible. Plastic bags may be used if the sample is too large. Submit whole fruit unless instructed otherwise.

Quality Assurance/Quality Control (QA/QC)

QA/QC samples are incorporated into vegetation/fruit (V/F) sampling investigations in order to enhance sample integrity, increase the confidence of analytical data, and to prevent reporting positives caused by contamination.

One type of QA/QC sample that can be incorporated into a V/F sampling study is an "extract split." It would be difficult to maintain homogeneity with a split field V/F sample. Instead, the sample is divided into two parts after the extraction has been completed by a chemist. One of the two extracts is analyzed at the primary lab; the other is sent to a second QC lab.

In the process of performing the analyses on the V/F field samples, the lab should also perform its own internal QC. When negotiating with the lab prior to submitting samples, request that they provide a copy of the internal QC performed with your samples and include this information in your report.

Shipping and Storage

If vegetation is to be analyzed for dislodgeable residue, samples should be shipped and stored at +4°C (wet ice/refrigeration). Indicate on the Chain of Custody that leaves should be saved so that the surface area may be determined. When a leaf punch is used, record the number and size of the punches on the paperwork.

If vegetation is to be analyzed for total residue, samples should be shipped at -70°C (dry ice), and stored at -10°C (freezer). Indicate on the Chain of Custody that the sample is to be analyzed for total residue.

In some instances it may be desirable to divide a total residue sample into a dislodgeable and internal residue analysis. In this case the sample is shipped and stored at +4°C (wet ice/refrigeration).

As with other residue samples, always use an ice chest that has never contained pesticides or diluted formulations.

VI. REFERENCE MATERIALS

Buchanan, T.J. and W.P. Somers. 1969. Discharge measurements at gaging stations. *In* Techniques of Water-Resources Investigations of the United States Geological Survey. Book 3, Chapter A8. 65 pg.

Biggar, J.W. and J.N. Seiber. 1987. Fate of Pesticides in the Environment. Proceedings of a Technical Seminar. Publication 3320. To order this publication, write to: Division of Agriculture and Natural Resources, University of California, 6701 San Pablo Avenue, Oakland, California 94608-1239

Keith, L.H. 1991. Environmental Sampling and Analysis: A Practical Guide. Lewis Publishers, Inc. 121 South Main Street, Chelsea, Michigan 48118.

APPENDICES

A. A Typical Chain Of Custody
B. Commissioner's Request for Analysis
C. Special Project Wells Chain of Custody
D. Equipment Sources
E. Numbering Water Wells in California (Department of Water Resources)
F. Routine Notifications When Pesticides are Confirmed in Drinking Water
G. Department of Water Resources - District Offices
H. Department of Water Resources Well Data, Form 429
I. Conversion Tables

APPENDIX - A

A TYPICAL CHAIN OF CUSTODY

APPENDIX A

CALIFORNIA STATE
DEPARTMENT OF
PESTICIDE REGULATION

CHAIN OF CUSTODY RECORD

(use ball point pen only)

ENVIRON. MONITOR. & PEST MGMT.
ENVIRON. HAZARDS ASSESSMENT
1220 N STREET, ROOM A-149
SACRAMENTO, CA 95814

30-034 (1/92)

Study #	Sample #	Date On					Date Off					Person Collecting	County #	Location Code	Sample Type
		Mo	Day	Yr	Time On		Mo	Day	Yr	Time Off					
1 2 3 4 5	6 7 8 9 10	11 12	13 14	15	16 17 18 19 20		21 22 23	24 25	26	27 28 29		30 31	32 33	34 35 36 37	38 39 40

Replicate #	Companion #	Flow Rate	Spray Sequence #	Sampling Interval	
41 42 43 44	45 46 47 48	49 50 51 52	53 54	55 56	57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

Partner: Location:

Machine #

Remarks

Key

Col 1
S = Spike

Col 2
* = Split

Col 38-40 (Sample Type)

LOV = Lo-Vol SOI = Soil
FOL = Foliage WAT = Water
FAL = Fallout FRU = Fruit
HIV = Hi-Vol TAN = Tank
DRO = Droplet Size

Lab Results (Save Extracts)

Chemical	Amount	Detection Limit

Extracted by: Extraction Date:
Analyzed by: Analysis Date:
Approved by: Report Date:

Task	Relinquished by	Received by	Date/Time
Container Prepared			
Collect/Transport			

Lab Name	Received for by lab	Date/Time	Logged in by	Date/Time	Lab #

Distribution: White to CDPR lab liaison, Yellow retained by lab, Pink to field files.

APPENDIX - B

COMMISSIONERS REQUEST FOR ANALYSIS

STATE OF CALIFORNIA
DEPARTMENT OF FOOD AND AGRICULTURE
Chemistry Laboratory Services

REQUEST FOR ANALYSIS AND REPORT OF ANALYSIS ON MATERIALS SUBMITTED BY COLLABORATING PUBLIC AGENCIES

531-002 (REV 3/80)

NOTICE: This form will be returned to you. Please type or print your address legibly with black ink and fill out form as completely as possible.

LABORATORY NO. _____

Date Received _____ (For Laboratory Use Only)

Agency Name

Address

City State Zip

cc Requester
Pesticide Enforcement (2)

(Please use address of collaborating agency only)

☐ Check if custody record is required

Sample consists of _____

Sample identification marks _____

Location/source of sample _____ County _____

Detailed description of problem _____

I hereby certify that the analysis requested is necessary in connection with matters relating to my official duties.

Sample Priority (From Back)
☐ # 1
☐ # 2
☐ # 3

Basis for Sample (Alleged Problem)
☐ Human Health Hazard
☐ Plant Symptoms or damage
☐ Animal/bee illness/loss
☐ Environmental Effects

By _____

Title _____

Date _____

State specific analysis requested

Laboratory Findings

Requested Disposition of Remaining Sample

☐ Results Phoned

By _____ Chemist

Date _____ Date

17077 150 12-80 CAM① OSP

Requester should mark each "Request for Analysis" form with the priority number that seems appropriate according to the priority descriptions shown below.

PRIORITY # 1: Those samples that deal with human or animal health or where remedial action can be taken to save crops. Examples that fall into this category are poisonings or pesticide drifts onto crops that are to be harvested soon. Samples given first priority will be started and completed within one day or as soon as possible. Requests will be telephoned with written reports to follow in the mail.

PRIORITY # 2: Those samples where information is needed but where no remedial action can be taken or where no immediate threat to human or animal health is present. Examples that fall into this category are bee kills or insecticide or herbicide drifts onto crops. These samples will be done on a routine basis with a goal of one week for completion. Results will be mailed routinely and results telephoned only if requested.

PRIORITY # 3: Those samples that have no pressing time factor involved. Examples that fall into this category are pesticide degradation studies or studies for pesticide use recommendations. These samples will be done as promptly as workload permits.

NOTE: The "Pesticide Enforcement Manual" published by the Department of Food and Agriculture contains information on proper sample size.

RECORD OF CUSTODY FOR REQUESTER SAMPLE NO.:

DATE	RECEIVED FROM	RECEIVED BY	ACTION TAKEN

APPENDIX - C

SPECIAL PROJECT WELLS CHAIN OF CUSTODY

APPENDIX C

CALIFORNIA STATE DEPARTMENT OF PESTICIDE REGULATION

30-032 (Rev. 5/94)

CHAIN OF CUSTODY RECORD
(use ball point pen only)

ENVIRON. MONITOR. & PEST MGMT.
ENVIRON. HAZARDS ASSESSMENT
1020 N STREET, ROOM 161
SACRAMENTO, CA 95814

Study #	Sample #	1 County	2 Well Number Twn	Rng	Sec	3 Sequence Number Tract	4 Meridian	5 D/Z Code	8 Date Sampled Mo	Day	Yr	Time	13 Lab Code	22 Well Use

Person Collecting

Partner

Minutes Pumped

Temp.

pH//adjusted pH

# of acid drops

Location Code

Well Owner's Name

18 Address #

Street

City

REMARKS:

Primary =

Backup =

Backup =

Backup =

Backup =

Field Blank =

LAB RESULTS:

10

(Save Extracts) 12

ST	CHEMICAL 9	11 AMOUNT p.p.b.	DETECTION LIMIT
☐	_____ ( )		
☐	_____ ( )		
☐	_____ ( )		
☐	_____ ( )		
☐	_____ ( )		
☐	_____ ( )		

20 19 14

Well Depth	Top Perf.	Bottom Perf.	P/N	Reserved	Analytical Method
				— —	

Extracted by: Extraction Date:

15

Analyzed by: Analysis Date:

Approved by: Report Date:

Task	Relinquished by	Received by	Date/Time
Container Preparation			
Collect/Transport			

Lab Name	Received for lab by	Date/Time	Logged In by	Date/Time	Lab #

Distribution: White to CDPR lab liaison, Yellow retained by lab, Pink to field files.

APPENDIX - D

EQUIPMENT SOURCES

EQUIPMENT SOURCES

AIR SAMPLER SUPPLIES

Kurz Instrument Inc.
P.O. Box 849
20 Village Square
Carmel Valley, California 93924
(800) 4-237350(800)4-AIRFLO

HI-Q Environmental Products Company
P.O. Box 2847
La Jolla, California 92038-2847

Anderson Samplers Inc.
4215 Wendell Drive
Atlanta, Georgia 30336
(800) 241-6898

SKC West
P.O. Box 4133
Fullerton, California 92634-4133
Inside California: (800) 228-4103
Outside California: (714) 992-2780

SOIL AND STREAM SAMPLING EQUIPMENT

Acker Drill Company, Inc.
P.O. Box 830
Scranton, Pennsylvania 18501
(717) 586-2061

Arts Manufacturing & Supply
(For soil augers)
105 Harrison
American Falls, Idaho 83211
(800) 635-7330

Forestry Suppliers, Inc.
(For soil probes & stream velocity meters)
205 West Rankin St.
Jackson, Mississippi 39204-0397
(800) 647-5368
(303) 433-7101

Geotech Environmental Equip. Inc.
(For water splitter)
1441 West 46th Avenue
Unit #17
Denver, Colorado 80211-2307

Wildco Sampling Equipment
Wildlife Supply Co.
(for aquatic sediment sampler)
301 Cass Street
Saginaw, Michigan 48602
(517) 799-8100

MISCELLANEOUS EQUIPMENT AND SUPPLIES

Consult the yellow pages of the nearest metropolitan area phone book for a list of scientific equipment supplies.

APPENDIX - E

NUMBERING WATER WELLS IN CALIFORNIA (DWR)

NUMBERING WATER WELLS IN CALIFORNIA

Need and Responsibility for Well Numbering

The need for a systematic and uniform procedure for numbering wells in California should become apparent when one realizes that (1) over 1,000,000 wells of all shapes, sizes, and condition are to be found in our State and on the average 10,000 to 25,000 more wells are added to this total each year; (2) records exist for more than 500,000 wells (i.e., construction logs, measurements of depth to water, physical, chemical and bacteriological, analyses of water, and pumping records); and (3) that a number of State, Federal County, City and local water agencies are involved in the development, use, and control of the water obtained from (or put into) these wells.

To prevent the uncoordinated numbering of wells by numerous agencies which would result in confusion and the preparation of erroneous information, a single agency is responsible for the assignment of well numbers. The Department of Water Resources (DWR) has that responsibility and authority.

The Well Numbering System

The State well numbering system is based on a rectangular system called the United States System of Surveying the Public Lands, commonly referred to as the Public Lands Survey, established by the Continental Congress in 1784. Under it all tracts of lands are tied to an initial point and identified as being in a township. A township is a square parcel of land six miles on each side. Its location is established as being so many six-mile units east or west of a north-south line running through the initial point (called the "principal meridian") and so many six-mile units north or south of an east-west line running through the point (called the "baseline"). In California there are three initial points and corresponding principal meridians and baselines. They are Mount Diablo, San Bernardino, and Humboldt, and we identify them by the letters M, S, and H respectively. The meridian lines parallel to, and east or west of, the principal meridian are called Range Lines. Lines parallel to, and north or south of, the baseline are known as Township Lines. Each township is described with respect to the initial point by its distance in numbers of six mile units and direction from that point i.e., north or south and east or west.

Every township is further divided into 36 parts called sections. A section is also described as a square parcel of land one mile on a side, each containing 640 acres. While this precision is customarily maintained, shortcomings in surveying techniques and the curvature of the Earth have resulted in some abbreviated or irregular sections. Spanish land grants which predate the public land surveys in California have not been subdivided in this manner. However, DWR in cooperation with the USGS has extended section lines on maps on which land grant boundaries appear and many extended section lines are published. DWR maintains an official file of these lines.

Such an existing grid system, familiar to agriculture, the real estate industry, surveyors and engineers is very useful for identifying "points on the ground" such as water wells. The State

well numbering system is an extension of the public land survey system and has been employed by DWR, USGS and other agencies for 50 years. Under it each well is assigned a unique number referred to as the State Well Number. The extension of the system involves dividing each section of land into sixteen 40-acre tracts. Once the well's location is established in the 40-acre tract it is assigned a sequence number. These sequence numbers are assigned in chronological order by DWR personnel. DWR maintains an index to prevent duplication.

Nomenclature and Notation: Examples

Following is an example of a State well number:

03S/04E-36N04S

Ignoring the slash and the hyphen the well numbers components are:

State Well Number	03S	04E	36	N	04	S
Township	/	/	/	/	/	/
Range		/	/	/	/	/
Section			/	/	/	/
40-Acre Tract				/	/	/
Sequence Number					/	/
Base and Meridian						/

Township is the third 36 square mile parcel of land (township) south of the initial point (T3S).

Range is the fourth 36 square mile parcel of land (township) east of the initial point (R4E).

Section is that parcel of land one mile square numbered 36 in township T3S/R4E.

Tract is that 40-acre parcel of land in section 36 lettered "N".

Sequence number 4 is the number assigned to this particular well in tract N of section 36 and it indicates that three other wells in this tract have been assigned numbers in the past.

Base and Meridian is that particular initial point, baseline and principal meridian to which this well is referenced, in this case S, the San Bernardino Base and Meridian.

How to Get Well Numbers Assigned

Agencies desiring well numbers should contact the district office of DWR in whose area the wells are situated. There are four district office locations; Red Bluff (Northern), Sacramento (Central), Fresno (San Joaquin), and Glendale (Southern). Addresses of these offices plus a list of the counties in each district area are attached. If you are uncertain as to which district office

to contact or you need additional assistance contact the Division of Local Assistance in Sacramento.

Your request for a State Well Number should be accompanied by:

1. A map of reasonably large scale or a sketch map showing the location of the wells with respect to prominent manmade features or natural landmarks and the distance to them. A most useful map is the standard USGS seven and one-half minute quadrangle topographic map with a scale of 1:24000 (2-1/2 inches = one mile).

2. An exact description of each individual well location including:

   a. Address of the property (county, city or town, street or highway address).

   b. If used, the name or number assigned to the well by its owner. (Agencies owning more than one well commonly identify each well by some designation.)

   c. The township, range, and section (if known).

   d. Direction and distance from the nearest city or town, roads, streets, canals, powerlines, or other identifier.

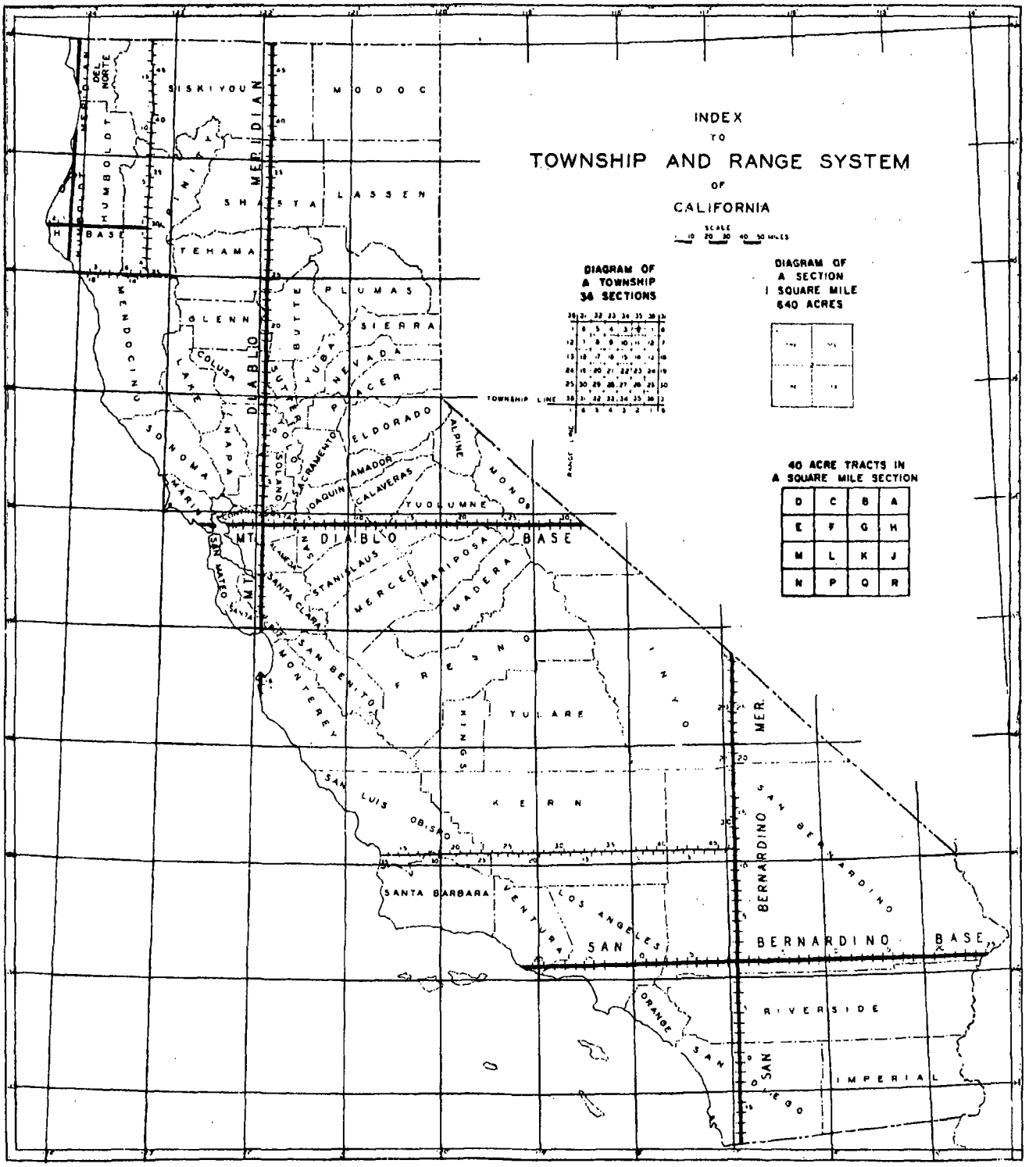
   e. Its location with respect to existing wells (distance and direction).

3. A description of the well itself, i.e., anything that is known about the well:

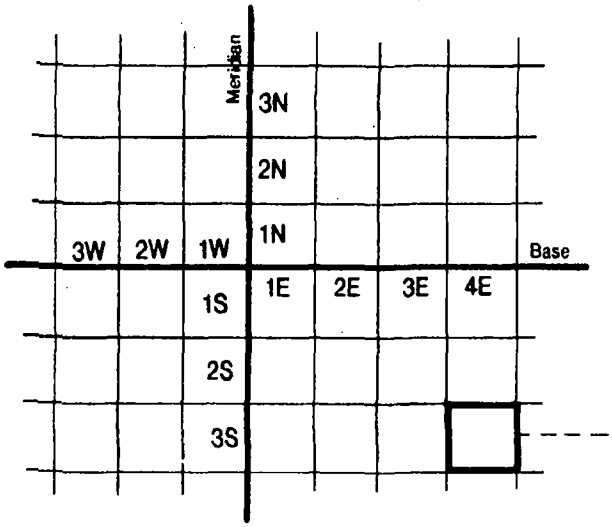
   owner
   date of construction (reconstruction or modification)
   driller
   depth of well
   casing material and its diameter
   pump horsepower and manufacturer of pump and motor including serial number
   utility company meter number

It is recognized that all this information may not be readily available for each well but the more there is the less the possibility of misnumbering and confusion at a later date.

DWR has for many years used a standardized form (DWR Form 429 "Well Data") for recording information for each well. Copies are available on request.


INDEX
TO
TOWNSHIP AND RANGE SYSTEM
OF
CALIFORNIA
SCALE
10 20 30 40 50 MILES
DIAGRAM OF A TOWNSHIP 36 SECTIONS
TOWNSHIP LINE
RANGE LINE
DIAGRAM OF A SECTION 1 SQUARE MILE 640 ACRES
40 ACRE TRACTS IN A SQUARE MILE SECTION
D C B A
E F G H
M L K J
N P Q R
HUMBOLDT BASE
HUMBOLDT MERIDIAN
DEL NORTE
SISKIYOU
MODOC
SHASTA
LASSEN
TEHAMA
MENDOCINO
GLENN
BUTTE
PLUMAS
SIERRA
COLUSA
LAKE
YUBA
NEVADA
PLACER
SONOMA
NAPA
SOLANO
SACRAMENTO
EL DORADO
ALPINE
MONO
AMADOR
CALAVERAS
JOAQUIN
YOLO
YUOLUMNE
MT DIABLO BASE
MT DIABLO MERIDIAN
SAN MATEO
SANTA CLARA
STANISLAUS
MERCED
MARIPOSA
MADERA
SAN BENITO
MONTEREY
FRESNO
KINGS
TULARE
INYO
KERN
SAN LUIS OBISPO
SANTA BARBARA
VENTURA
LOS ANGELES
SAN BERNARDINO MER.
SAN BERNARDINO BASE
ORANGE
RIVERSIDE
SAN DIEGO
IMPERIAL

STATE WELL NUMBER 03S/04E-36N04S



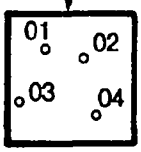
SAN BERNARDINO BASE
AND MERIDIAN
Township and Range
Numbering System

6	5	4	3	2	1
7	8	9	10	11	12
18	17	16	15	14	13
19	20	21	22	23	24
30	29	28	27	26	25
31	32	33	34	35	36

TOWNSHIP 03 SOUTH,
RANGE 04 EAST
Section Numbering System

D	C	B	A
E	F	G	H
M	L	K	J
N	P	Q	R

SECTION 36
Tract Numbering System



TRACT "N"
Well Numbering System
and Location

APPENDIX - F

ROUTINE DPR NOTIFICATIONS WHEN PESTICIDES ARE DETECTED IN GROUND WATER

Routine DPR Notifications When Pesticides Are Detected in Ground Water

The following parties are notified routinely when an active ingredient or breakdown product of a pesticide has been detected by DPR in a ground water sample collected from a well:

Parties	Notifications	
	Privately Owned Domestic and Irrigation Wells	Public Water Systems
Well Owner	Yes	Yes
County Agricultural Commissioner	Yes	Yes
County Environmental Health	Yes	Yes
County Health Officer	Yes	Yes
California Department of Health Services, Office of Drinking Water: Technical Programs Branch, Chief		Yes
California Department of Health Services, Office of Drinking Water: District Engineer		Yes

APPENDIX - G

DEPARTMENT OF WATER RESOURCES - DISTRICT OFFICES

DEPARTMENT OF WATER RESOURCES

DISTRICT OFFICES

NORTHERN DISTRICT
Post Office Box 607
Red Bluff, CA 96080
(2440 Main Street)
(916) 527-6530

Butte
Colusa
Del Norte
Glenn
Humboldt
Lake
Lassen
Modoc
Plumas
Shasta
Siskiyou
Tehama
Trinity

CENTRAL DISTRICT
3251 "S" Street
Sacramento, CA 95816-7017
(916) 322-7164

Alameda
Alpine
Amador
Calaveras
Contra Costa
El Dorado
Marin
Mendocino
Mono* (north of Mono Lake)
Napa
Nevada
Placer
Sacramento
San Francisco
San Joaquin
San Mateo
Santa Clara
Santa Cruz
Sierra
Solano
Sonoma
Sutter
Yolo
Yuba

SAN JOAQUIN DISTRICT
3374 E. Shields Avenue
Fresno, CA 93726-6990
(209) 445-5481

Fresno
Kern* (from Sierra Nevada west)
Kings
Madera
Mariposa
Merced
San Benito
Santa Cruz
Stanislaus
Tuolumne
Tulare
Monterey

SOUTHERN DISTRICT
Post Office Box 29068
Glendale, CA 91209-9068
(818) 543-4600

Imperial
Kern* (east of Sierra Nevada)
Los Angeles
Mono* (from Mono Lake south)
Orange
Riverside
Santa Barbara
San Bernardino
San Luis Obispo
Ventura

HEADQUARTERS
Division of Local Assistance
1020 Ninth Street
Sacramento, CA 95814
(916) 327-8861

*** Counties are located in two districts.**

APPENDIX - H

DEPARTMENT OF WATER RESOURCES - WELL DATA, FORM 429

STATE OF CALIFORNIA
THE RESOURCES AGENCY
DEPARTMENT OF WATER RESOURCES

State No. _____

WELL DATA

DISTRICT _____

Owner _____ | State No. _____
Address _____ | Other No. _____
Tenant _____
Address _____

Type of Well: Hydrograph ☐ Key ☐ Index ☐ Semiannual ☐

Location: County _____ Basin _____ No. _____

U.S.G.S. Quad. _____ Quad. No. _____

_____ ¼ _____ ¼ Section _____, Twp. _____, Rge. _____ MD SB H Base & Meridian

Description _____

Reference Point description _____

which is _____ ft. above / below land surface. Ground Elevation _____ ft.

Reference Point Elev. _____ ft. Determined from _____

Well: Use _____ Condition _____ Depth _____ ft.

Casing, size _____ in., perforations _____

Measurements By: DWR ☐ USGS ☐ USBR ☐ County ☐ Irr. Dist. ☐ Water Dist. ☐ Cons. Dist. ☐

Chief Aquifer: Name _____ Depth to Top Aq. _____ Depth to Bot. Aq. _____

Type of Material _____ Perm. Rating _____ Thickness _____

Gravel Packed? Yes ☐ No ☐ Depth to Top Gr. _____ Depth to Bot. Gr. _____

Supp. Aquifer _____ Depth to Top Aq. _____ Depth to Bot. Aq. _____

Driller _____

Date drilled _____ Log, filed _____ open (1) _____ confidential (2) _____

Equipment: Pump, type _____ make _____

Serial No. _____ Size of discharge pipe _____ in.

Power, Kind _____ Make _____

H. P. _____ Motor Serial No. _____

Elec. Meter No. _____ Transformer No. _____

Yield _____ G.P.M. Pumping level _____ ft.

Water Analysis: Min. (1) _____ San. (2) _____ H.M. (3) _____

Water Levels available: Yes (1) _____ No _____

Period of Record: Begin _____ End _____

Collecting Agency: _____

Prod. Rec. (1) _____ Pump Test (2) _____ Yield (3) _____

SKETCH

REMARKS

Recorded by: _____

Date _____

DWR 429 (Rev. 4/70)

APPENDIX - I

CONVERSION TABLES

TABLES OF WEIGHTS AND MEASURES

Linear Measure

1 inch	=		= 2.54	centimeters
12 inches	=	1 foot	= 0.3048	meter
3 feet	=	1 yard	= 0.9144	meter
5½ yards or 16½ feet	=	1 rod (or pole or perch)	= 5.029	meters
40 rods	=	1 furlong	= 201.17	meters
8 furlongs or 1,760 yards or 5,280 feet	=	1 (statute) mile	= 1,609.3	meters
3 miles	=	1 (land) league	= 4.83	kilometers

Square Measure

1 square inch			= 6.452	square centimeters
144 square inches	=	1 square foot	= 929	square centimeters
9 square feet	=	1 square yard	= 0.8361	square meter
30¼ square yards	=	1 square rod (or square pole or square perch)	= 25.29	square meters
160 square rods or 4,840 square yards or 43,560 square feet	=	1 acre	= 0.4047	hectare
640 acres	=	1 square mile	= 259	hectares or 2.59 square kilometers

---

Cubic Measure

1 cubic inch			= 16.387 cubic centimeters
1,728 cubic inches	=	1 cubic foot	= 0.0283 cubic meter
27 cubic feet	=	1 cubic yard	= 0.7646 cubic meter
		(in units for cordwood, etc.)	
16 cubic feet	=	1 cord foot	
8 cord feet	=	1 cord	= 3.625 cubic meters

Chain Measure

(for Gunter's, or surveyor's chain)

7.92 inches	=	1 link	= 20.12	centimeters
100 links or 66 feet	=	1 chain	= 20.12	meters
10 chains	=	1 furlong	= 201.17	meters
80 chains	=	1 mile	= 1,609.3	meters
		(for engineer's chain)		
1 foot	=	1 link	= 0.3048	meter
100 feet	=	1 chain	= 30.48	meters
52.8 chains	=	1 mile	= 1,609.3	meters

Apothecaries' Fluid Measure

1 minim		= 0.0038 cubic inch	= 0.0616 milliliter
60 minims	= 1 fluid dram	= 0.2256 cubic inch	= 3.6966 milliliters
8 fluid drams	= 1 fluid ounce	= 1.8047 cubic inches	= 0.0296 liter
16 fluid ounces	= 1 pint	= 28.875 cubic inches	= 0.4732 liter

---

Circular (or Angular) Measure

60 seconds (") = 1 minute (')
60 minutes = 1 degree (°)
90 degress = 1 quadrant or 1 right angle
4 quadrants or 360 degrees = 1 circle

Avoirdupois Weight

(The grain, equal to 0.0648 gram, is the same in all three tables of weight)

1 dram or 27.34 grains		= 1.772	grams
16 drams or 437.5 grains	= 1 ounce	= 28.3495	grams
16 ounces or 7,000 grains	= 1 pound	= 453.59	grams
100 pounds	= 1 hundredweight	= 45.36	kilograms
2,000 pounds	= 1 ton	= 907.18	kilograms

In Great Britain, 14 pounds (6.35 kilograms) = 1 stone
112 pounds (50.80 kilograms) = 1 hundredweight
2,240 pounds (1,016.05 kilograms) = 1 long ton.

Troy Weight

(The grain, equal to 0.0648 gram, is the same in all three tables of weight)

3,086 grains	= 1 carat	= 200	milligrams
24 grains	= 1 pennyweight	= 1.5552	grams
20 pennyweights or 480 grains	= 1 ounce	= 31.1035	grams
12 ounces or 5,760 grains	= 1 pound	= 373.24	grams

Apothecaries' Weight

(The grain, equal to 0.0648 gram, is the same in all three tables of weight)

20 grains	= 1 scruple	= 1.296	grams
3 scruples	= 1 dram	= 3.888	grams
8 drams or 480 grains	= 1 ounce	= 31.1035	grams
12 ounces or 5,760 grains	= 1 pound	= 373.24	grams

THE METRIC SYSTEM

Linear Measure

10 millimeters	= 1 centimeter	=	0.3937	inch
10 centimeters	= 1 decimeter	=	3.937	inches
10 decimeters	= 1 meter	=	39.37	inches or 3.28 feet
10 meters	= 1 decameter	=	393.7	inches
10 decameters	= 1 hectometer	=	328 feet 1 inch	
10 hectometers	= 1 kilometer	=	0.621	mile
10 kilometers	= 1 myriameter	=	6.21	miles

Square Measure

100 square millimeters	= 1 square centimeter	=	0.15499	square inch
100 square centimeters	= 1 square decimeter	=	15.499	square inches
100 square decimeters	= 1 square meter	=	1,549.9	square inches or 1.196 square yards
100 square meters	= 1 square decameter	=	119.6	square yards
100 square decameters	= 1 square hectometer	=	2.471	acres
100 square hectometers	= 1 square kilometer	=	0.386	square mile

Land Measure

1 square meter	= 1 centiare	=	1,549.9	square inches
100 centiares	= 1 are	=	119.6	square yards
100 ares	= 1 hectare	=	2.471	acres
100 hectares	= 1 square kilometer	=	0.386	square mile

Volume Measure

1,000 cubic millimeters	= 1 cubic centimeter	=	.06102	cubic inch
1,000 cubic centimeters	= 1 cubic decimeter	=	61.02	cubic inches
1,000 cubic decimeters	= 1 cubic meter (the unit is called a *stere* in measuring firewood)	=	35.314	cubic feet

Capacity Measure

10 milliliters	= 1 centiliter	=	.338 fluid ounce
10 centiliters	= 1 deciliter	=	3.38 fluid ounces
10 deciliters	= 1 liter	=	1.0567 liquid quarts or 0.9081 dry quart
10 liters	= 1 decaliter	=	2.64 gallons or 0.284 bushel
10 decaliters	= 1 hectoliter	=	26.418 gallons or 2.838 bushels
10 hectoliters	= 1 kiloliter	=	264.18 gallons or 35.315 cubic feet

Weights

10 milligrams	= 1 centigram	=	0.1543	grain
10 centigrams	= 1 decigram	=	1.5432	grains
10 decigrams	= 1 gram	=	15.432	grains
10 grams	= 1 decagram	=	0.3527	ounce
10 decagrams	= 1 hectogram	=	3.5274	ounces
10 hectograms	= 1 kilogram	=	2.2046	pounds
10 kilograms	= 1 myriagram	=	22.046	pounds
10 myriagrams	= 1 quintal	=	220.46	pounds
10 quintals	= 1 metric ton	=	2,204.6	pounds

Surveyor's (Square) Measure

625 square links	= 1 square pole	=	25.29	square meters
16 square poles	= 1 square chain	=	404.7	square meters
10 square chains	= 1 acre	=	0.4047	hectare
640 acres	= 1 square mile or 1 section	=	259	hectares or 2.59 square kilometers
36 square miles	= 1 township	=	9,324.0	hectares or 93.24 square kilometers

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Nautical Measure

6 feet	= 1 fathom	= 1.829 meters
100 fathoms	= 1 cable's length (ordinary) (In the U.S. Navy 120 fathoms or 720 feet = 1 cable's length; in the British Navy, 608 feet = 1 cable's length.)	
10 cables' lengths	= 1 nautical mile (6.076.10333 feet, by international agreement in 1954)	= 1.852 kilometers
1 nautical mile (Also called geographical, sea, or air mile, and, in Great Britain, Admiralty mile.)	= 1.1508 statute miles (the length of a minute of longitude at the equator)	
3 nautical miles	= 1 marine league (3.45 statute miles)	= 5.56 kilometers
60 nautical miles	= 1 degree of a great circle of the earth	

Dry Measure

1 pint		= 33.60 cubic inches	= 0.5505 liter
2 pints	= 1 quart	= 67.20 cubic inches	= 1.1012 liters
8 quarts	= 1 peck	= 537.61 cubic inches	= 8.8096 liters
4 pecks	= 1 bushel	= 2,150.42 cubic inches	= 35.2383 liters

1 British dry quart = 1.032 U.S. dry quarts

According to United States government standards, the following are the weights avoirdupois for single bushels of the specified grains: for wheat, 60 pounds; for barley, 48 pounds; for oats, 32 pounds; for rye, 56 pounds; for corn, 56 pounds. Some States have specifications varying from these.

Liquid Measure

1 gill	= 4 fluid ounces (see next table)	= 7.219 cubic inches	= 0.1183 liter
4 gills	= 1 pint	= 28.875 cubic inches	= 0.4732 liter
2 pints	= 1 quart	= 57.75 cubic inches	= 0.9463 liter
4 quarts	= 1 gallon	= 231 cubic inches	= 3.7853 liters

The British imperial gallon (4 imperial quarts) = 277.42 cubic inches = 4.546 liters. The barrel in Great Britain equals 36 imperial gallons, in the United States, usually 31½ gallons.

1.0 BACKGROUND

This standard operating procedure (SOP) details all procedures for using the Geoprobe System, a hydraulically operated sampling probe, and its specialized sampling tools. The procedures described within this SOP include soil gas sampling, groundwater sampling, and soil sampling procedures as well as procedures for installing piezometers and vapor sampling implants. This SOP also describes general procedures for rod removal, backfilling, and decontamination which are common elements to all sampling procedures. This SOP No. 054 replaces former draft SOP No. 054 (Geoprobe Soil Gas Sampling) and draft SOP No. 055 (Geoprobe Groundwater Sampling).

Use of the Geoprobe System is only one of many sampling techniques used by Tetra Tech EM Inc. (Tetra Tech); however, it is a preferred sampling method when certain conditions prevail. Specifically, Geoprobe sampling should be considered when sampling is limited to relatively shallow depths and any of the following are factors: (1) costs must be kept very low, (2) the time period is short to perform the sampling, (3) maneuverability is important, and (4) the required sampling volume is limited.

Prior to the use of the Geoprobe equipment, all buried utility lines and other underground structures must be marked because this equipment can penetrate buried piping and tanks. A diagram of the Geoprobe system is shown in Figure 1.

1.1 PURPOSE

The purpose of SOP No. 054 is to establish positioning, preparing, and sampling procedures; piezometer and vapor sampling implant installation procedures; rod removal procedures; backfilling procedures; and decontamination procedures to guide field personnel.

1.2 SCOPE

The procedures outlined in SOP No. 054 are applicable to all Tetra Tech personnel involved in soil gas, soil, or groundwater sampling using the Geoprobe System or any of its specialized equipment. It also is applicable to all personnel using the Geoprobe System to install piezometers and vapor sampling implants. This SOP, in fact, applies to all uses of the Geoprobe System.

1.3 DEFINITIONS

Because Geoprobe Systems is a corporation specializing in an innovative sampling process, many of the terms used to describe its equipment are specialized and specific. For this reason, familiarity with hydraulic system, soil sampling, soil gas sampling, and groundwater sampling terms is necessary. These terms are discussed below.

1.3.1 Hydraulic System Terms

The following terms are principally used to discuss the basic operation of the hydraulic punch and its major components. If terms are encountered while using this SOP that are not listed below, check Sections 1.3.2, 1.3.3, and 1.3.4 below.

Hydraulic Punch: The principal part of the Geoprobe System, the hydraulic punch, looks very much like a small mobile drilling rig and is usually attached to a truck or van. The punch's hydraulic system uses the weight of the vehicle for support and a hydraulic system installed in the vehicle to advance sampling tools into the soil (see Figure 1).

Hammer: The hydraulic hammer pounds the rods and accessories into the soil once the hydraulic punch is unable to push it farther (see Figure 1).

Control Panel: The control panel is located near the hydraulic punch and contains the levers that control the movement of the punch (see Figure 2).

Probe Lever: This lever is found on the control panel and causes the hydraulic punch to push the drive rod and accessories into the soil. Overall, this lever controls the vertical movement of the punch (see Figure 2).

Hammer Lever: This lever is found on the control panel and engages the hydraulic hammer when the hammer release valve is moved to its extended position (see Figure 2).

Hammer Release Valve: This lever is found on the front of the hydraulic punch and allows the hammer to work when in its extended position. If the valve is not extended, pushing the hammer lever will not engage the hammer.

Foot Lever: This lever is found on the control panel and lowers the foot of the hydraulic punch so that it rests on the ground to stabilize the punch (see Figure 2).

Extend Lever: This lever is found on the control panel and controls the horizontal movement of the hydraulic punch. The lever extends the punch out of the van or truck. It also enables the hydraulic punch to extend about 2 feet from the rear of the vehicle (see Figure 2).

Fold Lever: This lever is found on the control panel and folds and unfolds the hydraulic punch so that it can be easily moved and stored (see Figure 2). This lever enables the hydraulic punch to move from the horizontal position to the vertical position.

Electrical Control Switch: This switch is found on the control panel and turns on the Geoprobe System's hydraulic system. None of the other levers work until this switch is turned on. It has slow, fast, and off speed positions (see Figure 2).

Vacuum System Panel: The vacuum system panel is located near the right rear of the vehicle and contains the vacuum system controls, the hydraulic oil cooling switch, and the remote ignition (see Figure 2).

Remote Ignition: This device is found on the vacuum system panel and allows one to start the vehicle's engine from near the hydraulic punch instead of walking around the vehicle and climbing into the vehicle's cab (see Figure 2).

Hydraulic Oil Cooling Switch: This switch is found on the vacuum system panel and turns on the auxiliary cooling system for the hydraulic oil (see Figure 2).

Vacuum/Volume (Vac/Vol) Pump Switch: This switch is found on the vacuum system panel and allows pressure to build up in the vacuum tank (see Figure 2).

Vacuum Line Valve: This valve is found on the vacuum system panel and opens and closes the vacuum line (see Figure 2).

Sample Line Gauge: This gauge is found on the vacuum system panel and registers the sample line pressure in inches of mercury (see Figure 2).

Drive Rod: The Geoprobe drive rod (sometimes called a probe rod) is a high-strength-steel, hollow tube with a 1-inch outer diameter. Though the rods come in 1-foot, 2-foot, and 3-foot lengths, the standard length is 3 feet. Each rod is threaded on both ends and has a male end and a female end (see Figure 3).

Drive Cap: This cap is a steel cap screwed onto the male end of the drive rod so that the rod can be pushed or hammered into the soil without damaging its threads. The drive cap is always installed to the top of the drive rod before advancing probe rods or sampling tools (see Figure 3).

Pull Cap: This cap is a steel cap that screws onto the male end of the drive rod and is used to pull the drive rod from the soil once the sample has been collected (see Figure 3).

Anvil: This piece of steel is placed inside the hydraulic punch at the point where the hammer actually makes contact. The anvil transfers the force of the hammer to the drive cap (see Figure 3).

Rotary-Impact Carbide-Tipped Drill Bit: This 18-inch or 24-inch steel drill bit fits directly into the hydraulic punch and is used to drill through concrete or hard asphalt. The bit does not spin with appreciable torque but is driven by the hammer, spinning only slightly to clear itself of debris (see Figure 3).

Chain-Assisted Pull Cap: This modified pull cap is attached to the hydraulic punch with a chain. It is most useful when the drive rod, for one reason or another, is not aligned directly underneath the hydraulic punch. With this cap, the rod can still be pulled using the punch (see Figure 3).

Rod Extractor: This tool threads onto a drive rod and is sent down into the hole made by a drive rod that has broken in the soil. The rod extractor, which looks a little like a drill bit, is then hammered into the broken rod and is used to pull the broken rod from the soil (see Figure 3).

Rod Pull Plate: This steel plate has a hole in its center through which a drive rod can be fitted. It is used to extract drive rods when installing piezometers, soil gas implants, or to expose the screen to groundwater when using a screen point sampler (see Figure 3).

O-Ring: An O-ring is a rubber ring used to seal sections of drive rods or various other Geoprobe tools so that, once together, they are air- and water-tight.

Teflon Tape: This inert, sticky tape can be used to create air-tight seals when pieces of the drive rod or accessories are threaded together. The tape can replace an O-ring.

1.3.2 Soil Sampling Terms

These terms are usually used when discussing soil sampling using the Geoprobe System. Sometimes, though, the terms are used when discussing other sampling techniques. If terms are encountered while using this SOP that are not listed below, check Sections 1.3.1 above and Sections 1.3.3 and 1.3.4 below.

Shelby Tube: This tube is used to collect large samples of cohesive soils. Its greatest disadvantages are that it cannot be used to sample from depths greater than about 10 feet and has no mechanism to stay closed until reaching the proper depth (see Figure 4).

Shelby-Tube-Drive Head: This 2-inch diameter piece of steel attaches to the Shelby tube using hex bolts. The Shelby-tube-drive head consists of two parts: a standard 2-inch Shelby tube drive head and a Geoprobe drive rod adapter. This allows the 2-inch wide Shelby tube to be driven by the hydraulic punch, which is actually designed for 1-inch diameter drive rods (see Figure 4).

Hex Bolts: These are the bolts used to attach a Shelby tube to a drive head (see Figure 4).

Extruder Latch: This device secures the Shelby tube to the extruder rack during the extrusion process that removes the soil from the tube (see Figure 4).

Extruder Piston: This piston is threaded onto a drive rod, and with the help of the hydraulic punch, extrudes the soil sample from the Shelby tube (see Figure 4).

Probe-Drive Systems: This sampling system allows samples to be collected at deeper depths than the Shelby tube system. Each probe-drive sampler remains closed until it reaches the depth desired and then is opened by those operating the punch by removing a stop pin (see Figure 5). The sampler is then pushed through the soil at the desired depth and removed. Three types of probe-drive samplers exist: the standard sampler, the Kansas sampler, and the large bore probe-drive sampler.

Standard Probe-Drive Sampler: This probe-drive sampler has a diameter of 1 inch and lengths of 10 or 24 inches. Its greatest difference from the other probe-drive sampler is that it does not have a removable cutting shoe (see Figure 5).

Stop Pin: This pin stops the point of a probe-drive sampler from retracting into the sampler tube. Once it is removed, the sample can be collected (see Figure 5).

Piston Rod: This rod connects the drive head of a probe-drive sampler to the sampler's point. Once the stop pin is removed, this rod slides through the sampler, allowing the point to retract inside the tube (see Figure 5).

Drive Head: This head is the top of a probe-drive sampler, which allows the piston rod to slide straight up the sample tube after the piston stop has been removed and the drive rod is advanced (see Figure 5).

Cutting Shoe: This portion of the probe-drive sampler cuts through the soil once the point is allowed to retract inside. The Kansas samplers and large-bore sampler have removable cutting shoes (see Figure 5).

Extruder Rack: This device holds soil samplers in place during extrusion. The Shelby tube extruder rack is shown in Figure 4, and the standard probe-drive extruder rack is shown in Figure 5.

Extension Rod: This long, thin, threaded, solid rod is dropped through a drive rod to the probe-drive sampler so that the stop pin can be removed. Often more than one extension rod (an extension rod string) must be put together to reach the stop pin (see Figure 5).

Extension Rod Handle: This small metal handle screws to the top of the extension rod string so that it can be turned easily while being used to remove the stop pin (see Figure 5).

Large-Bore Probe-Drive Sampler: This probe-drive sampler is 1-1/8 inches in diameter and 24 inches long. Its larger width allows for the collection of larger samples. The diameter also allows for acetate or brass liners to be used in sample collection. These liners can make viewing the sample easier and preparing it for analysis simpler.

Kansas Sampler: This specially designed probe-drive sampler has a removable cutting shoe to enable easy extraction of soil and to allow the shoe to be replaced without replacing the complete sampler.

Kansas Stainless Sampler: This sampler has a stainless-steel sampling tube. It works in the same way as the Kansas sampler.

1.3.3 Soil Gas Sampling Terms

The following terms are used principally to discuss soil gas sampling. A few terms, though, are used while discussing groundwater sampling as well. If unfamiliar terms not listed below are encountered while using this SOP, check Sections 1.3.1 and 1.3.2 above and Section 1.3.4 below.

Expendable Point: These points fit into an expendable point holder that has been threaded into the lead drive rod. When the drive rod is pulled back, these points do not move with it, leaving a gap from which soil gas can be collected. The points are ultimately left in the ground (see Figure 6).

Expendable Point Holder: This holder threads into the leading drive rod. It is used for driving expendable points (see Figure 6).

Retractable Point Holder: This holder lifts off its point, leaving a gap so that soil gas can be drawn, but unlike expendable points, the holder does not separate completely and ultimately is retrieved with the lead drive rod (see Figure 6).

Gas Sampling Cap: When using the standard soil gas sampling method, the gas sampling cap replaces the drive cap on top of the drive rod and allows tubing to be connected to the drive rod. A soil gas sample is drawn through the probe rod through this cap and into a sample container (see Figure 6).

Post-Run Tubing (PRT) System: This system collects soil gas drawn directly through a tube instead of through the drive rod itself. The system involves one of two specially designed point holders, each threaded on top so that an adapter that has been attached to the tube can be screwed into it after being advanced down the drive rod string. The two point holders differ in that one uses a retractable point and the other uses an expendable point (see Figure 7).

PRT Expendable Point Holder: This holder is threaded into the leading probe rod and is used for driving expendable points (see Figure 7).

PRT Adapter: The PRT adapter attaches the tubing through which the soil gas is to be drawn to the point holder, which has been driven to the proper sampling depth (see Figure 7).

Polyethylene Tubing: This tubing is the preferred tubing for connecting the PRT system to the sample container. Its stiff nature, however, sometimes makes it difficult to attach to the sample container and a coupler of Tygon tubing is necessary (see Figure 7).

Tygon Tubing: This tubing is the preferred tubing for connecting soil gas sampling containers to the drive rod and vacuum system. It often is also necessary as a coupler sample between the stiff polyethylene tubing used with PRT sampling systems and the sample container.

Glass Bulb: This bulb of glass has valves on each side and a neoprene septum through which gas can be withdrawn. The bulb is used to collect soil gas and can be used as the container in which the gas is taken for analysis (see Figure 8).

Tedlar Bag: This small bag has a valve on it. It is placed in an air-tight chamber, the air in the chamber is evacuated, and the bag fills with soil gas. The bags can then be taken for analysis.

Tedlar Bag Chamber: Tetra Tech uses these modified, air-tight kitchen containers as vacuum chambers. These chambers are modified with nipples on each side, which enable it to be attached to a vacuum pump, to a Tedlar bag, and to the Tygon tubing.

1.3.4 Groundwater Sampling Terms

The following terms are used to discuss groundwater sampling. If unfamiliar terms not listed below are encountered while using this SOP, check Sections 1.3.1, 1.3.2, and 1.3.3 above.

Mill-Slotted Well Point: This 3-foot long tube has 15 mill-cut slots in it, each 2 inches long and 0.020 inches wide. Only the bottom 2 feet of this tube is slotted, and sometimes mill-slotted well points come in two parts: a 2-foot slotted section and a 1-foot unslotted section. The slots allow groundwater to enter (see Figure 9).

Geoprobe Screen Point Sampler: This sampler has a 19-inch screen that encases a perforated stainless-steel sleeve. Once in place, the screen allows the water to enter the tube and prevents coarse sediment from entering the tube (see Figure 9).

Thieving Tube: This tube is used to extract the water from either mill-slotted well points or Geoprobe screen point samplers, Tetra Tech uses polyethylene tubing as thieving tubes. This tubing is lowered into the water, capped on top, and then extracted. The result is much like putting a straw into a glass of water, sealing the straw with a finger and lifting it. This method is used primarily for the collection of groundwater samples to be analyzed for volatile organic compounds. A check valve can also be attached to the thieving tube which seals the bottom and holds the groundwater within the tube.

Check Valve: This stainless steel valve has a small ball which, when attached to a thieving tube, floats to the top of the groundwater table and then sinks, ultimately sealing the thieving tube with groundwater. Oscillating the thieving tube will allow groundwater to rise within the tube for larger retrieval volume.

Well Mini-Bailer: This specially designed bailer drops through the drive rods and into the groundwater in the mill-slotted well point or screen point. A small ball in the bailer floats to the top and then sinks, ultimately sealing the bailer after it fills with about 40 milliliters of groundwater.

1.4 REFERENCES

The following references were used to prepare this SOP:

Driscoll, F.G. 1987. *Groundwater and Wells.* Second Edition. Johnson Division. St. Paul, Minnesota.

Fisher Scientific. 1991. "The Fisher Catalog of Scientific Instruments."

Geoprobe Systems. 1990. "8-M Operations Manual." July 27.

Geoprobe Systems. 1991. "Accessory Tools Catalog."

Geoprobe Systems. 1992. "Equipment and Tools Catalog."

2.0 POSITIONING, PREPARING AND SAMPLING PROCEDURES

The Geoprobe System uses a hydraulic punch that is usually installed in the back of a van or truck to first push and then to hammer its hollow drive rod through soils. Depending on which tools are attached to the end of the drive rod and which sampling equipment is attached to it, the Geoprobe can be used to remove soil, soil gas, or groundwater. It can also be used to drill through cement or concrete and can aid in the installation of piezometer wells and vapor sampling implants. The following sections detail the procedures for positioning the Geoprobe unit, preparing the sampling system, and sampling with the Geoprobe unit.

2.1 POSITIONING THE GEOPROBE UNIT

Before the Geoprobe System can be used, the Geoprobe hydraulic punch and accessories must be properly positioned near the sampling site. The hydraulic punch and other equipment also needs to be prepared. In cases where concrete or other hard surfaces hinder sampling, the Geoprobe must be used to reach soil. This section details methods to perform these activities.

To position and unload the Geoprobe System use the following procedures:

1. Drive the vehicle containing the Geoprobe System to the sampling location and align the center of the rear of the vehicle with the point at which the sample will be taken. The rear bumper should be 1 to 2 feet from the sampling point so that the foot of the hydraulic punch can be extended out over it.

2. Shut off the vehicle.

3. Put it in park.

4. Set the emergency brake before proceeding.

5. One person only should operate the hydraulic punch and the assembly and disassembly of probe rods and accessories. A second person is usually necessary to handle the samples and to decontaminate equipment. All personnel present must wear steel-toed shoes, gloves, and eye protection. When drilling through concrete or using the hydraulic hammer, ear protection is also necessary.

6. Once ready to take the sample, start the engine using the remote ignition located in the right rear of the vehicle. As a safety device, the remote ignition will not work unless the vehicle is in park.

7. Activate the hydraulic system by turning on the electrical control switch. The vehicle's engine must be running for the hydraulic system to work.

8. Slowly extend the Geoprobe out of the vehicle using the extend lever. Always use the slow speed on the hydraulic controls when positioning the hydraulic punch. The punch and mast should be far enough out of the van or truck so that the mast will not strike the roof when it is unfolded.

9. Unfold the hydraulic punch out of the vehicle using the fold lever. Once the punch has been lined up perpendicular to the ground surface, lower the foot of the punch using the foot lever until the vehicle itself is raised about 1 foot on its springs. This stabilizes the vehicle and punch. **Never lift the vehicle completely off the ground using the foot lever.** Doing so destabilizes the vehicle and hydraulic punch and may cause damage to equipment or injury to those nearby. Also, as pressure is placed on the rod, tools, and accessories, the foot of the punch may begin to lift. Do not allow it to lift farther than 6 inches from the ground. Allowing it to lift farther than 6 inches may throw the vehicle off balance and cause the rod to bend or break.

The Geoprobe System is now positioned. If it is necessary to drill through concrete or hard asphalt, use the following procedures:

1. Raise the hydraulic punch using the probe lever and then deactivate the hydraulic system by turning the electrical control switch to off. The hydraulic system should always be turned off when the hydraulic controls are not being used.

2. Place the drill bit into the hydraulic hammer. The bit is not used with a drive rod or anvil.

3. Activate the hammer rotation control knob, which is located on the hydraulic hammer, by turning the knob counter-clockwise. This allows the drill bit to rotate when the hammer lever on the control panel is pressed.

4. Activate the hammer release valve, which is located on the hydraulic hammer, by pulling the lever out and down.

5. To drill through solid surfaces, both the probe and hammer mechanisms of the hydraulic punch must be used. The hammer mechanism drives the drill bit in a percussion fashion and causes it to turn slightly. The probe mechanism allows the hammer and bit to be raised and lowered so that the bit can clear itself of debris. Once ready to begin, turn on the hydraulic system.

6. Fully depress the hammer lever. This lever needs to remain depressed throughout the drilling procedure and keeps the bit pounding and rotating.

7. Put pressure on the bit by pressing the probe lever down. Using this lever, advance the bit in small increments through the concrete or other hard surface. If advanced too quickly, the bit will bind and stop rotating. Should this happen, raise the punch slightly to allow the bit to rotate. If too little pressure is placed on the bit, too little percussion will occur, and drilling will be slow.

8. Continue drilling, in small increments, until soil has been reached. At that time prepare for sampling.

2.2 PREPARING THE SAMPLING SYSTEM

Before the hydraulic punch is used to sample, decisions must be made concerning which type of sample will be taken, whether several samples will be taken at varying depths, and which type of Geoprobe sampling equipment will be used. The following sections discuss preparation procedures for soil sampling, soil gas sampling, and groundwater sampling.

2.2.1 Soil Sampling

The samplers attached to the hydraulic punch for soil sampling come in two forms. The first type is the 2-inch diameter Shelby tube system that is common to other soil sampling methods. The second system

uses various specially designed probe-drive systems that remain completely sealed while being pushed or driven to a particular depth. They then are opened to allow a sample to be collected. The Shelby tube and probe-drive systems are discussed below.

Shelby Tube System

The Shelby tube is a thin-walled steel tube, 2 inches in diameter and 30 inches long, with four mounting holes around its top. It allows large amounts of soil to be sampled at once, but the soil must be relatively cohesive. Because the tube remains open at all times, the tube cannot be driven to great depths and must be removed and replaced after coring 30 inches of soil. Usually, the Shelby tube system is chosen when large amounts of soil are needed at depths no deeper than 10 feet. Rocky or sandy soils are not conducive to this sampling method.

To prepare for sampling using Shelby tubes, use the following procedures:

1. First attach a Shelby tube to the Shelby-tube-drive head by putting the head's hex bolts through the holes in the tube.

2. Next, screw a Geoprobe drive rod adapter into the top of the drive head to allow the 2-inch-wide Shelby tube to be driven by the hydraulic punch and hammer, which are actually made for 1-inch outer diameter drive rods.

3. A drive cap is then screwed onto the top of Geoprobe drive rod adapter. The tube is now ready to be attached to the hydraulic punch.

4. To attach the tube, raise the hydraulic punch using the probe lever and then turn off the Geoprobe hydraulic system.

5. Lift the hammer latch and insert the anvil inside.

6. Place the assembled Shelby tube sampler so that it is aligned under the anvil.

The hydraulic punch is now ready to drive a Shelby tube and collect a sample core. For collecting soil cores at depths of greater than 30 inches, attach sections of probe rod to an assembled Shelby tube sampler and drive the sampler down the same hole using a new Shelby tube for each 30-inch increment in depth.

Probe-Drive Systems

All of the probe-drive systems work in essentially the same way. A sampler is attached to a hollow drive rod, inserted into the hydraulic punch, and punched or hammered into the soil. Once the sampler reaches the depth at which the sample is to be taken, a stop pin in the sampler is removed using an extension rod that has been dropped through the inside of the hollow drive rod. The release of the stop pin allows the point of the sampler to retract inside the sample tube as the sampler is further advanced into the soil. The probe is then punched through the soil where the sample is to be taken. The rod and probe are then pulled to the surface for sample extraction.

Currently, three types of samplers are used in the probe-drive systems: the standard probe-drive sampler, the Kansas sampler, and the large bore probe-drive sampler. Preparation of each is slightly different. Each is discussed separately below.

Standard Probe-Drive Samplers

The standard probe-drive sampler comes in 10- and 24-inch lengths. The proper length is determined by the size of the sample desired. The point of this sampler is connected to a piston rod that will slide through its length. At its top, the piston rod is connected to the drive head, which keeps it centered and holds the piston stop pin, which stops the piston from sliding.

To prepare the standard probe-drive sampler, use the following procedures:

1. Insure that the sampler is assembled and complete, and that the piston stop pin which is reverse threaded is tightly locked so that the sampler point will not slide into the sampling tube.

2. Attach a shortened Geoprobe drive rod to the sampler so that the total length is nearly the standard 3 feet. If the 10-inch sampler is used, a 2-foot drive rod should be attached, and if the 24-inch sampler is used, a 1-foot drive rod should be attached.

3. Screw a drive cap onto the top of the shortened drive rod. The sampler is now ready for attachment to the hydraulic punch.

4. To insert the probe-drive sampler, raise the hydraulic punch using the probe lever, and then turn the hydraulic system off.

5. Lift the hammer latch and insert the anvil inside.

6. Place the assembled standard probe-drive sampler and shortened drive rod directly under the anvil so that the drive cap touches the anvil and the point of the sampler is aimed at the place where the sample is to be taken. The standard probe-drive sampler and the hydraulic punch should both be vertical.

Kansas Samplers

The Kansas sampler is much like the standard probe-drive sampler. However, it has a removable hardened cutting shoe near its point that allows it to penetrate rockier soils and to be easily replaced and decontaminated. Kansas samplers come in two versions: the Kansas Stainless Sampler, which has a stainless-steel tube, and the Kansas Sampler, which has an alloy steel tube.

To prepare a Kansas sampler, use the following procedures:

1. Ensure that the hardened cutting shoe is in place.

2. Assemble and install the Kansas sampler in the same manner as the standard probe-drive sampler (see Procedures 2 through 7 above).

Large Bore Samplers

The large bore sampler, similar to both types of Kansas samplers, has a removable cutting shoe and works in the same manner. It is slightly larger than the Kansas samplers, usually 24 inches long and 1-1/8 inches wide. The larger bore allows for the use of acetate or brass liners. The soil, therefore, can be removed easily by removing the liner. The acetate liner allows for easy visual examination of the core and can be easily sliced away so that the sample can be prepared for the laboratory. The brass liners come in four 6-inch sections that allow for easy separation and packaging of 6-inch soil samples. Some laboratories accept full 6-inch brass liners, allowing the samples to be collected with a very minimal disturbance to the soil matrix.

To prepare a large-bore sampler, use the following procedures:

1. Place the desired liner into the sampler by unscrewing the cutting shoe and sampler drive head from the two ends and then inserting the liner.

2. Assemble the sampler and attach a 12-inch drive rod to the sampler.

3. Screw a drive cap onto the top of the drive rod.

4. Place the assembled sampler and drive rod under the hydraulic punch in the manner detailed in the section above for preparing standard probe-drive samplers (see Procedures 5, 6, and 7 above).

2.2.2 Soil Gas Sampling

Two main methods are used to collect soil gas using the Geoprobe system: the standard method and the PRT system.

To use the standard method, the drive rods are decontaminated and assembled in an air-tight manner as they are punched into the soil. To ensure an air-tight seal, either Teflon tape or an O-ring can be placed on the male threads of the drive rods. The probe rods are driven approximately 6 inches below the area from where the sample is to be taken. The rods are then lifted approximately 6 inches leaving the expendable point and a small opening between the point and the end of the rod behind. A gas sampling cap is then attached to the top of the rod, a vacuum pump removes the necessary volume of gas, and the sample is collected.

To collect soil gas samples using the PRT system, polyethylene tubing attached to a stainless steel adapter is pushed through the drive rod after the rod is in place. The tubing and adapter is then reverse threaded onto the top of the PRT expendable point holder, and the gas is collected through the tubing. This method increases the accuracy of soil gas sampling, eliminates the potential for leaks in the rod, and simplifies probe rod decontamination.

Standard Method

Only decontaminated drive rods can be used with the standard method. Rods should be decontaminated using the procedures in Section 6.0 of this SOP.

To prepare a decontaminated drive rod for soil gas sampling using the standard method, use the following procedures:

1. Screw an expendable point holder into the female end of a 3-foot drive rod. (Note: a retractable point can also be used with this method; however, decontamination requirements almost always preclude its use.)
2. Place an expendable point into this holder.
3. Screw a drive cap onto the male end of the drive rod.
4. Place the rod into the hydraulic punch.
5. Turn on the hydraulic system.
6. Install the anvil within the hydraulic punch's hammer by lifting the hammer latch and inserting it.
7. Place the assembled drive rod directly under the anvil so that the drive cap faces the anvil and the expendable point is aimed at the desired sampling location.
8. Push sampler and hydraulic punch through the soil to gather the sample.

PRT System

Two types of PRT systems are available. The first uses an expendable point holder and expendable point like the standard method. The second uses a retractable point holder that lifts off of the drive-point without actually separating from it. Both systems allow the threading of a PRT adapter and tubing through the drive rod so that the gas can be taken from the depth required without being sucked through the drive rod.

To prepare the drive rod and sampler for PRT soil gas sampling, use the following procedures:

1. Select the desired PRT sampler (either one with an expendable point or one with a retractable point) and ensure that the PRT adapter easily screws into the threads on top of the sampler. This step is necessary to ensure that the adapter will fit easily when it is affixed from above ground.
2. If using the sampler with an expendable point, attach the point.

3. Screw the sampler to the end of a shortened drive rod so that the total length of the sampler is nearly 3 feet.

4. Screw the drive cap to the other end of the drive rod.

5. Attach the drive rod and sampler to the hydraulic punch using the same procedures detailed in the standard method (see Procedures 4, 5, and 6 above).

2.2.3 Groundwater Sampling

The Geoprobe System offers two systems for collecting groundwater, each with several groundwater sampling options. The first method involves the use of a mill-slotted well point. The second method uses a specially designed Geoprobe screen point sampler.

Mill-Slotted Well Points

The mill-slotted well point is a 2- or 3-foot length of hollow steel tubing with 15-millcut slots in it, each 2 inches long and 0.020 inches wide. Once in place, groundwater enters the tube through these slots. To prepare the mill-slotted well point, use the following procedures:

1. Screw a solid drive point into the female end of the sampler.

2. If a 2-foot well point is being used, screw the sampler to a 1-foot length of drive rod.

3. Screw a drive cap to the other end of the well point or 1-foot drive rod.

4. Place the sampler and rod into the hydraulic punch by raising the punch as much as necessary and turn hydraulic system off.

5. Install the anvil within the hydraulic punch's hammer by lifting the hammer latch and inserting it.

6. Place the mill-slotted well point sampler under the anvil with the drive cap near the anvil and the point aimed at the sampling location.

Geoprobe Screen Point Sampler

The Geoprobe screen point sampler has a 19-inch screen encased in a perforated stainless-steel sleeve. The screen remains encased in the sleeve until the screen point sampler reaches the desired depth. The

rod is then pulled back approximately 19 inches, leaving the screen exposed to the formation. Flexible tubing can be pushed through the drive rod and attached to the sampler using the adapters for the PRT soil gas system, enabling groundwater to be removed without touching the drive rod. Decontaminating the drive rod is subsequently easier.

To prepare a Geoprobe screen point sampler, use the following procedures:

1. Close the screen on the sampler.
2. Attach its expendable point.
3. Attach the sampler to a shortened drive rod so that the assembly is nearly 30 inches long.
4. Place the sampler into the hydraulic punch using the methods detailed for mill-slotted well points (see Procedures 4, 5, and 6 above).

2.3 SAMPLING

Sampling procedures for the Geoprobe hydraulic punch are similar for all samplers and sampling media. This section presents general procedures that apply to all samplers and sample types, and specific operating procedures for soil, soil gas, and groundwater.

2.3.1 General Procedures

All control panel switches have a slow and fast position. All switches should initially be set at the slow position when positioning the punch and the sampling tools. In all cases, the hydraulic system should be shut off when not in operation and when adapters and additional drive rods are put into place. The hydraulic punch should be turned off any time it is not actually in operation.

The Geoprobe hydraulic punch is designed with a key safety feature that will shut it off if the controls are released. If the operator senses that something is wrong, he or she must release the controls and stop operating the punch until all is well. At no time should the foot of the punch be allowed to lift higher than 6 inches off the ground because the punch will destabilize and may bend the drive rod or sampling tube.

Also, at no time should part of a human body be placed on top of a drive cap while the cap is near the anvil or under the foot of the hydraulic punch.

Once the assembled sampler or drive rod is under the anvil, both it and the hydraulic punch should be vertical. Positioning the drive rod and sampler is critical in order to drive the rod vertically. Not positioning the sampler or drive rod vertically will result in problems when attaching subsequent drive rods needed to reach the proper depth and with rod retrieval.

To begin probing in soils of normal texture, use the following procedures:

1. Activate the hydraulic punch and push down on the probe lever on the control panel so that the probe slowly lowers itself. Always use the slow control on the first rod or sampler.

2. Continue to press on the probe lever until the rod or sampler is completely forced into the soil. The point of the rod will then be nearly 3 feet into the soil.

Soils and other materials are often too hard for the hydraulic punch's probe mechanism to penetrate. When this occurs, the hammer on the hydraulic punch should be used in accordance with the following procedures:

1. Ensure that the hammer rotation valve is closed.

2. Use the hydraulic punch to put pressure on the rod, sampler, and soil. When the probe rod refuses to move, the foot of the hydraulic punch will begin lifting off the ground. Never allow the foot to lift more than 6 inches off the ground, but never use the hammer with the foot resting on the ground surface.

3. If the probe foot lifts off the ground, the hydraulic punch may no longer be perpendicular. If this occurs, use the machine's fold lever, which is located on the control panel, to correct the punch's position.

4. Press the hammer lever on the control panel. The rod should now advance. Never use the hammer unless there is downward pressure on the drive cap because doing so may damage the equipment.

5. Stop hammering periodically and check to see if the probe rods can be advanced using the probe mechanism only.

When samples are to be taken at depths of greater than 3 feet, additional drive rods must be added to those already in the ground. Shelby tube soil sampling procedures for adding rods are discussed in Section 2.3.2. For all other sampling methods, use the following procedures to add drive rods:

1. Using the probe lever, raise the hydraulic punch off the portion of the drive rod protruding from the ground.
2. Unscrew the drive cap from the drive rod.
3. If using the standard method of collecting soil gas or other sampling methods that will draw the sample through the length of the entire drive rod, wrap the threads of the drive rod with Teflon tape or push an O-ring over the threads to make the drive rod string air- and water-tight.
4. Screw another drive rod onto the first drive rod protruding from the ground. Tighten the rods together with a pipe wrench.
5. Screw a drive cap onto the top of the new drive rod.
6. Place the hydraulic punch over the new drive rod and push the rod farther into the ground.

As the rod string is pushed farther into the ground, it will sometimes begin to loosen. The rods should remain tight so that the threads are not damaged. Occasionally, stop probing and twist the rod string with a pipe wrench to ensure that all of the joints remain tightly sealed.

2.3.2 Soil Sampling

This section presents procedures used to sample soils using either the Shelby tube sampling method or any of the probe-drive systems. In all cases, sampling tools should never be advanced farther than their length once they are opened because the sampler will overfill. If the sampler overfills, it could be damaged or expand, causing it to fall off the drive head.

Shelby Tube Sampling Procedures

Because the Shelby tube does not remain closed until it reaches the desired sampling depth and because it is not connected to a drive rod but to a Shelby drive head, sampling procedures for Shelby tubes differ greatly from soil sampling with other methods. New drive rods cannot be continuously added. Sampling

at depths of greater than 30 inches requires a step-like procedure. For example, to sample to a depth of 90 inches, three Shelby tubes are needed. The first is advanced from 0 to 30 inches and then removed. The second is pushed through the hole made by the first and advanced to a depth of 60 inches and removed. The third is also pushed through the 60-inch deep hole and advanced from 60 to 90 inches.

Samplers must be ready to change sampling methods if necessary. For example, if soils are not cohesive, they tend to drop out of the Shelby tube as it is pulled from the ground. Also, if the soils are not cohesive, they tend to collapse into the hole left by the initial tube before the second and third tubes can be pushed into place. For this reason, use of the Shelby tube method is impractical at depths of greater than 10 feet. Rocky soils are also difficult to sample with a Shelby tube sampler because they tend to destroy the sampler while it is being driven into the ground.

To sample using the Shelby tube method, use the following procedures:

1. Turn on the hydraulic system and slowly press the Shelby tube into the soil using the probe lever on the control panel.

2. Once the tube has reached the sampling depth or has been extended to nearly its full 30-inch length, stop the hydraulic punch and raise it off the drive cap and Shelby tube drive head.

3. Unscrew the drive cap.

4. Screw on a pull cap.

5. Lower the hydraulic punch and lift the hammer latch. Remove the anvil. Place the latch around the pull cap so that the latch will hold the cap to the hydraulic hammer.

6. Using the probe lever, raise the hydraulic punch to pull the Shelby tube from the ground.

If the desired sampling depth is greater than 30 inches, additional Shelby tubes and probe rods must be used. The tubes are then prepared for probing using the methods presented in Sections 2.2.1 and 2.3.1 above. To advance the Shelby tube deeper, the tubes are pushed through the hole left by the first tube using the method detailed above.

Once a Shelby tube core has been retrieved from a sampling point, it must be extruded from the Shelby tube sampler using the following procedures:

1. Lower the hydraulic punch using the probe lever so that its mast will not strike the top of the van as it is folded.

2. Lift the foot of the hydraulic punch using the foot lever.

3. Slowly and carefully fold the hydraulic punch using the fold lever.

4. Once the punch is horizontal, the Shelby tube extruder bracket can be placed onto the punch's foot. This bracket will hold the Shelby tube in place and allow the punch to push the soil out of the tube.

5. Screw an extruder piston onto a drive rod and a drive cap on the drive rod's other end.

6. Place the drive rod into place under the horizontal drive punch.

7. Place the full Shelby tube into the extruder rack and secure it with the extruder latch.

8. A pan or container should be held at the end of the Shelby tube to collect sample material as it is extruded.

9. The probe lever activates the hydraulic punch and pushes the soil from the Shelby tube.

Tetra Tech's SOPs on packaging and documenting samples, SOPs Nos. 016, 017, 018, and 019, should be used to prepare the sample for analysis.

Probe-Drive System Sampling Procedures

All three types of probe-drive samplers work in essentially the same way. The sampler is advanced to just before the proper sampling depth and then the drive point is released by removing a stop pin using solid extension rods that have been dropped through the hollow drive rod. The point is then pushed back into the body of the sampler as the sampler fills with the soil sample.

In addition to the general procedures listed in the Section 2.3.1, the probe must be stopped at just before the desired sampling depth so that the stop pin can be removed. Pushing the probe too far will require starting over.

To use the probe-drive sampling system to sample soil, use the following procedures:

1. Attach additional drive rods as discussed in the general procedures in Section 2.3.1.

2. Stop the hydraulic probe just before the desired sampling depth.

3. Raise the hydraulic punch, turn off the hydraulic system, and remove the drive cap.

4. Insert an extension rod into the drive rod and screw additional extension rods together until the assembly reaches the same depth as the sampler.

5. Attach a small extension rod handle to the top of the extension rod.

6. Rotate the extension rod handle clockwise until the leading extension rod has turned the stop pin and disengaged it.

7. Pull and unscrew each extension rod from the hollow drive rod. The stop pin should be attached to the bottom of the extension rod string. If not, repeat Procedures 1 through 6.

8. To sample, mark the drive rod with tape or chalk about 10 inches above the ground if a 10-inch sampler is used or 24 inches from the ground if a 24-inch sampler is used.

9. Replace the drive cap and start the hydraulic system.

10. Drive the rod until the tape or chalk mark touches the ground. Be careful not to overdrive the sampler. Doing so could compact the soil in the sampler or cause it to balloon outward, making soil removal and extrusion difficult.

11. Raise the hydraulic punch and replace the drive cap with the pull cap. Remove the anvil.

12. Latch the pull cap underneath the hydraulic hammer latch and pull the rods out of the ground, disassembling the rod as needed.

13. Check to ensure that a soil sample is now in the sampler.

Once a soil sample has been removed from the ground, it can be extruded using the Geoprobe. The tools supplied by Geoprobe Systems for extruding soil from probe-drive samplers do not require the Geoprobe to be folded and horizontal. If liners are used with large-bore samplers, extrusion is usually unnecessary. When extrusion is necessary for probe-drive samplers, use the following procedures:

1. Raise the foot of the hydraulic punch off the ground using the foot lever on the control panel.

2. Attach the extruder rack onto the foot of the punch so that its crossbeam rests on top of it.

3. Completely disassemble the sampler. In all cases, remove the piston, point, and drive head of the sampler. If using the Kansas and large-bore samplers, unscrew the removable cutting shoe as well.

4. Insert the sample tube into the extruder with its cutting end up.

5. Insert a disposable wooden dowel or the reusable steel piston above the soil and below the hydraulic punch so that pressure on the dowel or piston from the punch will push the soil out of the bottom of the sample tube.

6. Position proper sampling jars or trays under the sample tube and very slowly use the probe lever to force the soil out of the tube. Injury can result if the soil is quickly forced from the tube.

The soil sample is now ready for packaging or on-site laboratory analysis. For large-bore samplers, the soil may be contained in a plastic sleeve that can be sliced away once the soil is to be packaged or in a brass sleeve that may be capped on both ends and shipped to the laboratory as is. Tetra Tech's SOPs on packaging and documenting samples for analysis should be followed when collecting samples using the Geoprobe System.

2.3.3 Soil Gas Sampling Procedures

The standard method and the PRT system are used for collecting soil gas using the Geoprobe System. The standard method requires the drive rods to be sealed together with either O-rings or Teflon tape to ensure an air-tight seal so that soil gas from depths other than the bottom of the drive-rod string cannot penetrate the system.

The PRT system draws soil gas through continuous tubing that is dropped through the drive rod after the drive rod has reached the desired level. The tubing is then attached directly to the point holder at the end of the drive-rod string.

For both methods, the drive rod should be driven to the desired depth. The drive cap should be replaced by the drive pull cap, and the rod should be pulled back out of the hole approximately 6 inches. This 6-inch void is the area where the soil gas sample is collected from. A pipe wrench or vise-grip pliers should be attached to the pipe just above the foot of the hydraulic punch so that the wrench or pliers rests on the foot to stop the drive rod from working its way back down into the hole.

Tygon tubing should be replaced between each sample for both sampling methods to avoid cross contamination.

The standard method and the PRT system sampling procedures are presented below. In addition, procedures for collecting soil gas in Tedlar bags, glass bulbs, and adsorption tubes is also presented below.

Standard Method

To gather a sample using the standard method, raise the hydraulic punch as mentioned above and replace the drive cap with a gas sampling cap. This cap is designed to fit the drive rods and is used to connect them by tube to a vacuum supply. Once the tubing has connected the gas sampling cap to the vacuum supply, remove the volume of air necessary to ensure that none of the gas being drawn was in the rod during probing, and then collect the sample in either Tedlar bags, glass bulbs, or adsorption tubes as discussed below.

PRT System

To use the PRT system (with either an expendable or a retractable point) to collect soil gas samples use the following procedures:

1. Secure the PRT adapter to the end of a piece of polyethylene tubing 1 to 2 feet longer than the total length of the drive-rod string. The adapter must fit tightly within the tubing. If it does not, tape it into place. Also, ensure that the O-ring is in place on the threaded end of the adapter.

2. Remove the drive cap from the probing rod and lower the adapter into it, holding on to the tubing.

3. Grasp the excess tubing and apply downward pressure. Turn the tubing counter-clockwise to engage the adapter threads on the sampler holder.

4. Pull up lightly on the tubing to test engagement of threads. If the adapter has not engaged, try again. If it repeatedly does not engage, soil may have intruded into the drive rod either during probing or, in the case of the retractable point, when the rod was pulled back to leave the point opening. Use the threaded extrusion rods to clean out the threads.

5. In most cases, the adapter will easily screw into place. The sampler is now ready to collect samples in either Tedlar bags, glass bulbs, or adsorption tubes using the procedures presented below. After the sample is collected and the sampler and tube is removed from the ground, the O-ring should be checked to ensure that a good seal exists between the sampler and adapter. If the O-ring is tightly smashed, the seal should be good.

6. Discard polyethylene tubing and use new polyethylene tubing for each sample.

Tedlar Bags

Soil gas can be collected for chemical analysis in a 500-cubic-centimeter Tedlar gas sampling bag by inducing a vacuum on the exterior of the bag. The following procedures should be used to collect soil gas samples in Tedlar bags:

1. For the PRT system, connect a short (6- to 12-inch) piece of Tygon tubing to the free end of the polyethylene tubing protruding out of the drive rod. For the standard method, connect the Tygon tubing to the soil gas sampling cap.

2. Attach the other end of the Tygon tubing to one end of the Tedlar bag chamber. Tetra Tech uses modified, plastic, air-tight kitchen containers for these chambers. They are inexpensive and work well.

3. Connect another piece of Tygon tubing 2 feet to 3 feet long to the other end of the Tedlar bag chamber and to the nipple on the bottom of the vacuum system panel.

4. Place the lid on the Tedlar bag chamber.

5. Turn the vacuum/volume (vac/vol) pump switch on and allow pressure to build in the vacuum tank. Make sure that the vacuum line valve is closed before turning on the pump switch.

6. Open the vacuum line valve and purge three times the volume of ambient air out of the Tedlar bag chamber and PRT tubing or probe rods. The equations for determining purge volumes are as follows:

   Probe rods or tubing

   $$V = \pi r^2 H$$

   where

   V = Volume
   π = 3.14159
   r = Radius of tube or rod
   H = Length of tube or rod

Vacuum chamber

$$V = LWH$$

where

V = Volume
L = Length of chamber
W = Width of chamber
H = Height of chamber

7. Close the line valve.

8. Clamp the Tygon tubing shut with hemostats.

9. Remove the lid from the Tedlar bag chamber.

10. Connect a Tedlar gas sampling bag to the fitting inside the Tedlar bag chamber and open the valve on the gas sampling bag.

11. Place the lid back on the Tedlar bag chamber, seal it tightly, and remove the hemostats.

12. Turn the vac/vol pump switch on and open the vacuum line valve to create a vacuum in the chamber. The Tedlar bag should fill once the vacuum is created. The rate at which the Tedlar gas sampling bag fills depends on the permeability of the soil. The minimum amount of soil gas needed for analysis is approximately 0.5 liter. If less than 0.5 liter is collected after 4 minutes of sampling, raise the soil gas probe 0.5 foot and continue to evacuate the vacuum chamber for another minute. If the minimum required volume of soil gas is not collected, repeat the procedure. If the minimum required volume of soil gas is still not collected, abandon the collection process. All steps conducted should be accurately recorded in the logbook even if no samples are satisfactorily collected.

13. After the soil gas sample is collected in the Tedlar bag, clamp the Tygon tubing with hemostats.

14. Turn off the vacuum pump.

15. Remove the vacuum chamber lid.

16. Close the valve on the Tedlar gas sampling bag and remove the bag from the chamber. Label the Tedlar bag with the appropriate information.

Glass Bulbs

The following procedures should be used to collect soil gas in glass bulbs:

1. Turn the vac/vol pump switch on and allow pressure to build in the vacuum tank. Make sure that the vacuum line valve is closed before starting the vacuum pump. The inside scale of the vacuum tank gauge is calibrated in inches of mercury. The outside scale is calibrated for volume in liters (at standard temperature and pressure). Obtain the desired vacuum and turn the vacuum pump off.

2. Connect a short (6- to 12-inch) piece of Tygon tubing to the sample cap or PRT protruding from the drive rod.

3. Connect one end of the labeled glass bulb to the Tygon tubing.

4. Connect another piece of Tygon tubing 3 feet to 5 feet long to the other end of the glass bulb and to the nipple on the bottom of the vacuum system panel.

5. Open the two stopcocks on the glass bulb.

6. Turn off the vacuum pump.

7. Turn the vacuum line valve to its open position.

8. Purge three times the volume of ambient air within the rods, bulb, and tubing. Equations for figuring out volumes are presented in the Tedlar bag discussion.

9. Turn the vacuum line valve to its closed position. Allow the pressure in the sample train to equalize (the sample line gauge should read zero).

10. Close the stopcocks on the glass bulb.

11. Remove the glass bulb and label it with the appropriate information.

Adsorption Tubes

The following procedure should be used to collect soil gas in adsorption tubes:

1. Connect a short (6- to 12-inch) piece of Tygon tubing to the sample cap or PRT protruding from the drive rod.

2. Connect this piece of tubing to the nipple on the bottom of the vacuum system panel and purge three volumes of air from the drive rod or PRT system as described in the discussion of the Tedlar bag method.

3. Use hemostats to clamp the Tygon tubing attached to the drive rod or PRT.

4. Insert the adsorption tube between the Tygon tubing from the drive rod or PRT and the Tygon tubing attached to the vacuum system panel.

5. Remove the hemostats and draw the required volume of air through the adsorption tube.

6. Remove the adsorption tube and place the appropriate caps on the tube ends.

7. Clearly label package, and ship the samples as required by the laboratory or Tetra Tech and U.S. Environmental Protection Agency (EPA) SOPs.

Soil Gas Sampling Pointers

If the needle on the vacuum line valve does not move, the soil at the sampling depth may be saturated, pore space may be too tight to yield a sample, or sampling train may be plugged. If the needle moves back to zero very quickly, either the soil at the sampling depth is very permeable or a leak is present in the sampling train.

In some soils, the needle may return to zero very slowly. The time it takes for the needle to return to zero is called the "recovery" time. Recovery time should be noted for each sample taken. This information will allow relative comparison of soil permeability. Recovery times of greater than 10 minutes should be considered suspect. The effect of leakage in the sampling system increases with longer recovery times. After 10 minutes, the operator should consider either changing the sampling depth, location, or length of pullback from the sampling tip, or switching entirely from soil gas sampling to grab sampling and analysis of soil.

2.3.4 Groundwater Sampling

The two options for sampling groundwater using the Geoprobe System follow procedures similar to those presented in Sections 2.3.2 and 2.3.3 above. The sections below detail procedures for using mill-slotted well point samplers and Geoprobe screen point samplers to sample groundwater.

Mill-Slotted Well Point Sampler

Once the mill-slotted well point reaches groundwater, the water will begin to flow through the slots. When the sample is to be analyzed for volatile organic compounds, do not use a vacuum to suck groundwater from the drive rod. If the sample is to be analyzed for other parameters such as metals, semivolatiles, pesticides, or explosives, using a vacuum on the drive rod is acceptable. In all cases,

polyethylene tubing can be used as a thieving rod by lowering its end into the drive rod, capping or sealing the tube's top, and then removing it. The preferred method for collecting samples for volatile organic analysis is to use a well mini-bailer. To collect groundwater samples with a mini-bailer, use the following procedures:

1. Raise the hydraulic punch, turn off the hydraulic system, and remove the drive cap.
2. Lower a well mini-bailer into the drive rod until it reaches the bottom. As it reaches the bottom, the check ball on the bailer's end will float in the groundwater and then slowly sink to the bottom.
3. Allow a couple of seconds for the ball to sink and set.
4. Pull the well mini-bailer out of the drive rod. The bailer should contain about 20 milliliters of groundwater.
5. Package and document the samples in accordance with Tetra Tech SOPs No. 016, 017, 018, and 019, or a similar EPA-approved procedure.

If a bailer is not required and volatile organic samples are not being collected, a foot valve sampler, vacuum trap, or peristaltic pump can be used to collect samples. Once the sample has been removed and packaged, the mill-slotted well point can be removed and decontaminated.

Geoprobe Screen Point Sampler

The Geoprobe screen point sampler contains a screen and screen plug that allows water to enter the rod. To collect groundwater samples with a Geoprobe screen point sampler, use the following procedures:

1. Push the sampler below the depth necessary to reach groundwater.
2. Raise the hydraulic punch and replace the drive cap with a pull cap. Also, remove the anvil.
3. Latch the pull cap under the hammer latch, and use the probe lever to lift the drive rod about 18 inches. Because the sampler has a expendable point, the point should stay at the deepest depth, and the screen and screen connector should fall out of the bottom of the sampler. Sometimes, however, the screen stays within the sampler and is lifted the 18 inches with the drive rod.
4. To ensure that the screen is exposed, attach a vice grip or pipe wrench to the rod above the foot of the hydraulic punch and raise the hydraulic punch. Then remove the pull cap

and place an extension rod through the tubing to push the screen into place. Additional extension rods can be attached to reach the desired depth.

To remove the groundwater sample for volatile organic analysis, with a well mini-bailer, follow steps 1 through 5 under the mill-slotted well point section above. Tubing can be used as a thieving rod with or without a check valve to collect groundwater samples as well. If the sampler is supplied with the optional PRT expendable point holder, then a PRT adapter can be pushed through the drive rod and threaded into place by following the PRT system Procedures previously discussed. A vacuum trap system or peristaltic pump can then be used to withdraw the sample. The PRT system method, however, should never be used when the sample is to be analyzed for volatile organic compounds because it involves using a vacuum to remove the sample.

3.0 PIEZOMETER AND VAPOR SAMPLING IMPLANT INSTALLATION PROCEDURES

The Geoprobe System's ability to quickly probe into soil allows for easy installation of both piezometers and vapor sampling implants. Both installation procedures are discussed below.

3.1 PIEZOMETER INSTALLATION

Piezometers are tubes that extend to groundwater and enable easy sampling of groundwater on a routine basis (see Figure 10). In addition to installing the piezometer, piezometers must be protected from the weather and from contamination. A well-head protector must therefore be installed around them. In some soil types, preparing the well-head protector may be the first step to installing a piezometer. For this reason, the directions below should be read completely before beginning piezometer installation. If a post-hole digger is to be used for well-head protector installation, Procedure 5 should be performed first. The piezometer should then be advanced through this hole.

To install temporary or permanent piezometers, use the following procedures:

1. Use the hydraulic punch to drive the temporary casing to the desired piezometer installation depth. Use the general procedures outlined in Section 2.3.1 above for details on driving the piezometer casing. The different temporary casings that can be used are described below. Geoprobe Systems also manufactures special drive caps, expendable points, and pull caps that fit these types and sizes of pipe.

a) 1-7/16-inch outside diameter by 1-3/16-inch inside diameter, RW-flush threaded pipe can be used as a temporary casing. This casing can be driven to an approximately 25- to 30-foot depth. Two sizes of piezometer wells can be installed inside of the temporary casing: (1) 3/4-inch outside diameter by 1/2-inch inside diameter, polyvinyl chloride (PVC) pipe, or (2) 1-inch outside diameter by 3/4-inch inside diameter, PVC pipe.

b) 1-13/16-inch outside diameter by 1-1/2-inch inside diameter, EW-flush threaded pipe can be used as a temporary casing. This casing can be driven to an approximately 15- to 20-foot depth. Three sizes of piezometer wells can be installed inside of the temporary casing: (1) 3/4-inch outside diameter by 1/2-inch insider diameter PVC pipe, or (2) 1-inch outside diameter by 3/4-inch inside diameter, PVC pipe, or (3) 1-1/2-inch outside diameter by 1-inch inside diameter, PVC pipe.

c) 1-1/4-inch outside diameter by 1-inch inside diameter, NPT-threaded pipe can be used as a temporary casing. This casing can be driven to an approximately 25- to 30-foot depth. Only 3/4-inch outside diameter by 1/2-inch inside diameter, PVC pipe piezometer wells can be installed inside of the temporary casing. If using NPT-threaded pipe, couplers are needed to attach each section of pipe.

2. Once the piezometer casing is at the proper depth, remove the drive cap and install the selected size piezometer pipe inside of the temporary casing.

3. Using a pull plate, remove the temporary casing.

4. If the hole stays open, attempt to install a sand pack around the slotted portion of the piezometer, and then place dry granular bentonite on top of the sand pack as a seal. One foot of bentonite is recommended for a good seal.

5. Dig an 8-inch nominal-diameter hole around the piezometer pipe. This hole should extend to a depth of 1.5 to 2 feet. A post-hole digger can be used for this procedure if the hole is dug prior to driving the temporary casing. The bottom 6 inches of this hole should be filled with dry granular or slurry bentonite. The remainder of the hole should be filled with concrete. A steel, locking, aboveground or flush-mount well protector should be inserted into the wet concrete to provide well-head security. A concrete pad can also be constructed around the steel well-head protector.

3.2 VAPOR SAMPLING IMPLANT INSTALLATION

Figure 11 presents diagrams of vapor sampling implants. To install vapor sampling implants, first punch a drive rod to the desired depth using an expendable point holder and an expendable point. Once at the desired sampling depth, use the following procedures:

1. Disengage the expendable point and retract the probe rod about 1 foot by raising the hydraulic punch, replacing the drive cap with a pull cap, removing the anvil, latching the pull cap onto the hydraulic hammer using its latch, and raising the hydraulic punch again using the probe lever.

2. Lock the rod into place so that it does not sink back into the hole by using vice grip pliers or a pipe wrench.

3. Unlatch the pull cap and raise the hydraulic punch again, leaving room to work freely.

4. Remove the pull cap.

5. Attach appropriate stainless-steel tubing to the vapor implant. If tubing is precut, allow 48 inches more than the required depth of the implant.

6. Insert the implant and tubing down the inside diameter of the probe rods until it stops. Note the length of the tubing inserted to ensure that the desired depth has been reached. Allow the excess tubing to extend out of the drive rod's top.

7. Pour glass beads down the inside diameter of the probe rod using a funnel to create a permeable layer around the implant.

8. Use the tubing extending from the drive rod to stir the beads into place. Do not lift up on the tubing while doing so.

9. Position the remaining tubing through the hole on a rod pull plate, and then place the drive rod through that hole.

10. Attach the plate to the hydraulic punch using its chain and slowly pull the rod up another 18 to 24 inches. While the punch pulls the rod, push down on the tubing so that it stays in place.

11. Pour bentonite seal mixture down the inside diameter of the probe rod. Stir the mixture using the tubing as before. The initial mixture may also be topped with distilled water to initiate the bentonite seal depending on the site and on the role the vapor implant is to play.

12. Pull the drive rod from the hole using the probe rod pull plate already attached, and then plug the hole using granular bentonite or a bentonite slurry mixture.

The vapor sampling implant should now be in place and the stainless steel tubing connected to it should be protruding from the ground. The vapor implant tubing should be protected by a well-head protector in the same manner as the top of the piezometer. Procedure 5 in Section 3.1 describes well-head protector installation.

4.0 ROD REMOVAL PROCEDURES

Throughout the above discussions, it has occasionally been necessary to remove drive rods and samplers. The standard removal procedures involve raising the hydraulic punch, turning off the hydraulic system, replacing the drive cap with a pull cap, removing the anvil, and then latching the pull cap under the hammer latch. The hydraulic punch can then be used to pull the rod from the ground.

Two deviations to this procedure often occur. The first deviation is necessary when sampling tubes are to be left inside the hole as the drive rod is removed, especially when soil gas implants or piezometers have been installed. Because of the presence of these sampling tubes, a pull cap cannot be screwed onto the top of the drive rod. Instead, a rod pull plate is used. This plate is a piece of steel with a hole in it large enough for a drive rod to fit through it. The plate has a hook on one end. The tubing and rod are pushed through the plate, and the pull plate is attached to the latch on the hydraulic punch by a chain. As the punch pulls up, the plate shifts, and the inside of the hole binds on the rod. This binding usually holds the rod to the plate and results in the rod being pulled up as the punch is raised.

The second deviation occurs when the rods have not been pushed perpendicular to the ground. In these cases, a specially designed chain-assisted pull cap is used. This cap looks like a pull cap but has a chain on it that fits under the latch of the hammer. Once the cap is screwed to the drive rod and latched to the probe, raising the probe raises the rod.

In a few cases, drive rods break while in the ground. To retrieve these rods, a rod extractor is used. This extractor looks something like a drill bit and is screwed to the end of a probe rod. A hammer is then used to pound the extractor into the top of the broken rod. The extractor joins the broken rod to the second drive rod so that they can be pulled out together.

5.0 BACKFILLING PROCEDURES

Unless otherwise specified in the site-specific sampling plan, holes made by sampling with Geoprobe System tools are to be backfilled with dry, fine, granular bentonite. Water may be added to activate the bentonite. Tops of the holes may then be filled with soil or concrete as necessary for each particular site.

6.0 DECONTAMINATION PROCEDURES

Between holes, the probe rods and sampling tools must be decontaminated. Because no provisions for decontamination are included in the Geoprobe System, a separate decontamination station must be provided. A wire brush, a barrel brush for reaming out the rods, and soft brushes will clean sticky soil from the probe rods and sampling tools. Follow Tetra Tech SOP No. 002 decontamination procedures when sampling soil or groundwater.

When sampling for soil gas by the standard method, Geoprobe rods and samplers are heated approximately 15 to 20 minutes by a 100,000-British thermal unit heater until they are too hot to touch with the bare hand. They are then allowed to cool before reuse. Do not heat the rods too much or the rod metal will fatigue.

When sampling for soil gas by the PRT method, the probe rods do not have to be decontaminated. However, the PRT expendable point holder and PRT adapter do need to be decontaminated. They can be heated on the dash of the vehicle with the defrost system or scrubbed in Alconox and water. Equipment blank samples can be collected, if necessary, as part of the quality control process.

Sampling plans may have different decontamination requirements. Most plans also require rinsate sample collection as part of the quality control process.

FIGURE 1

GEOPROBE SYSTEM

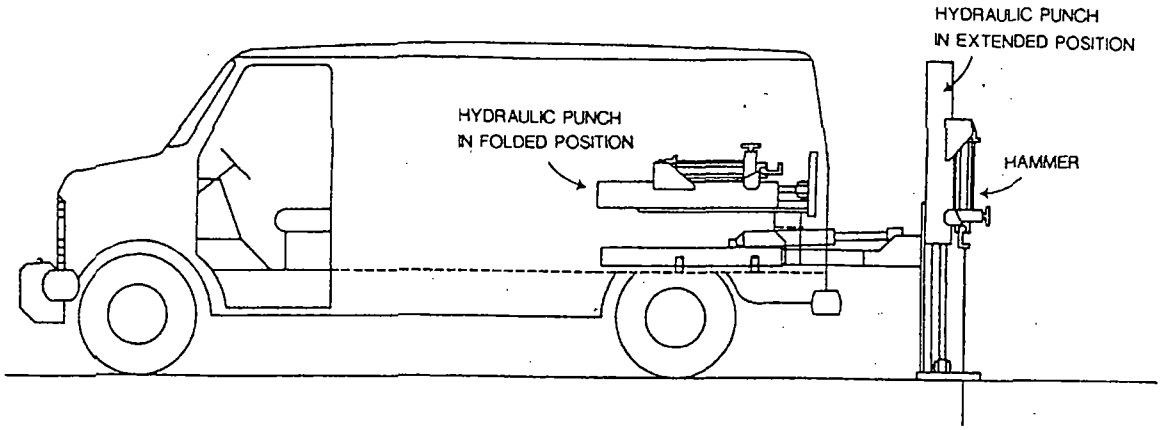
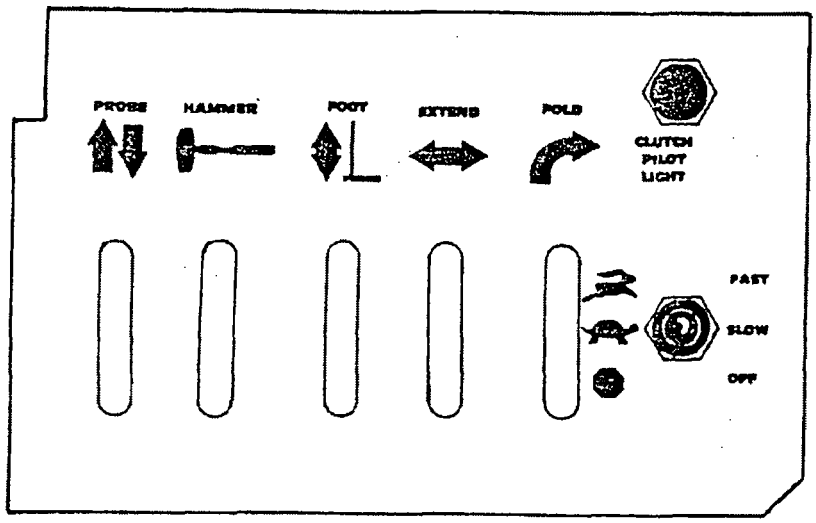
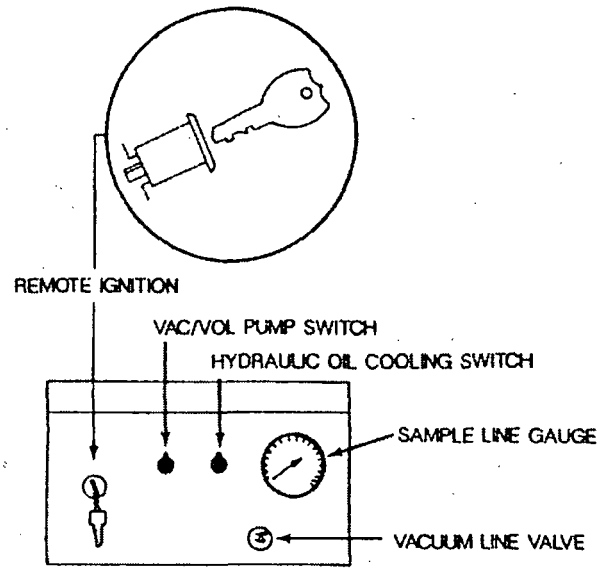


FIGURE 2

CONTROL AND VACUUM SYSTEM PANELS



CONTROL PANEL



VACUUM SYSTEM PANEL

FIGURE 3

GENERAL ACCESSORY TOOLS

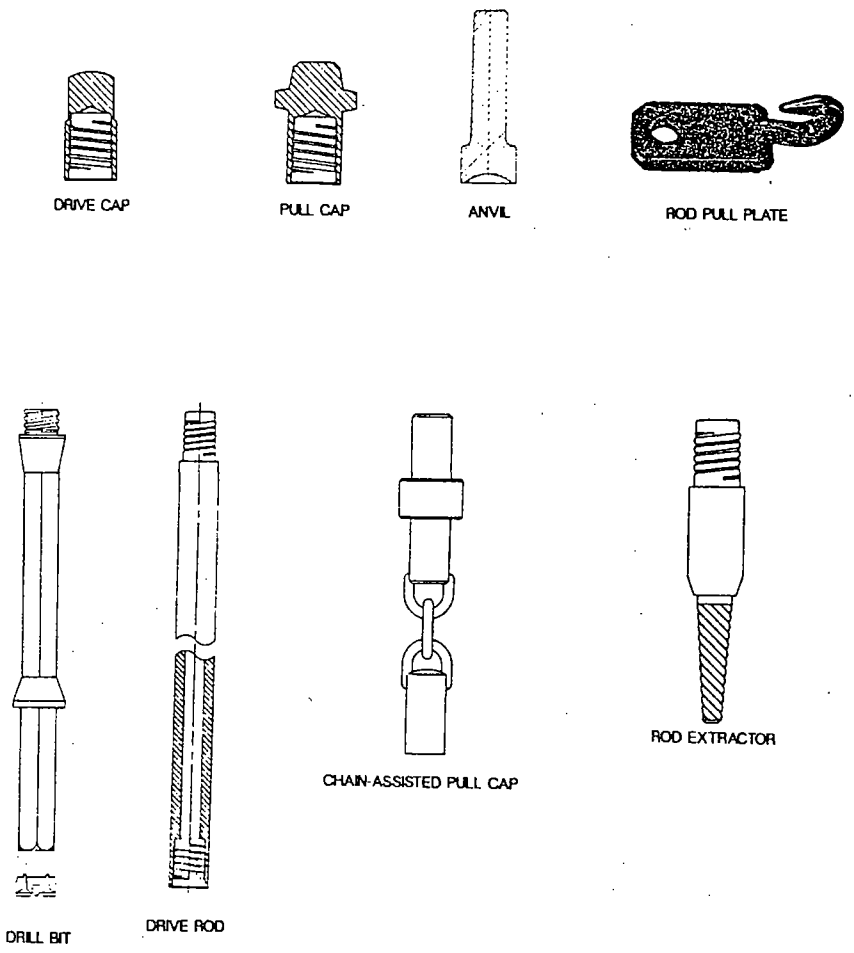
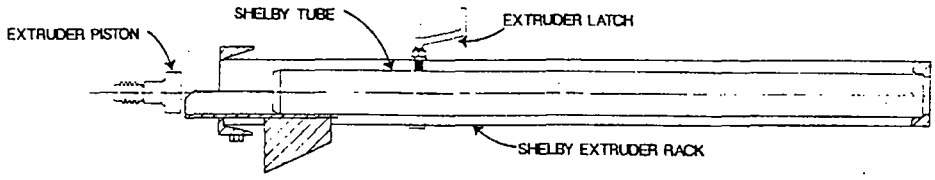


FIGURE 4

SHELBY TUBE ACCESSORIES



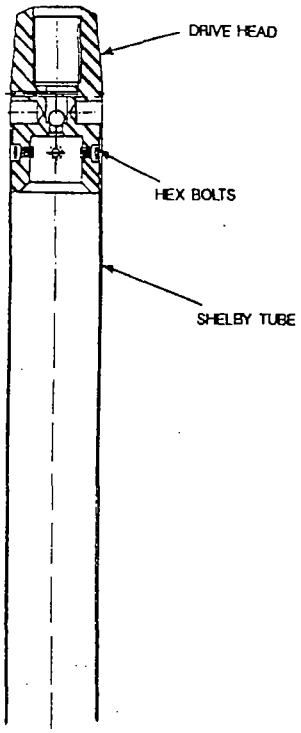
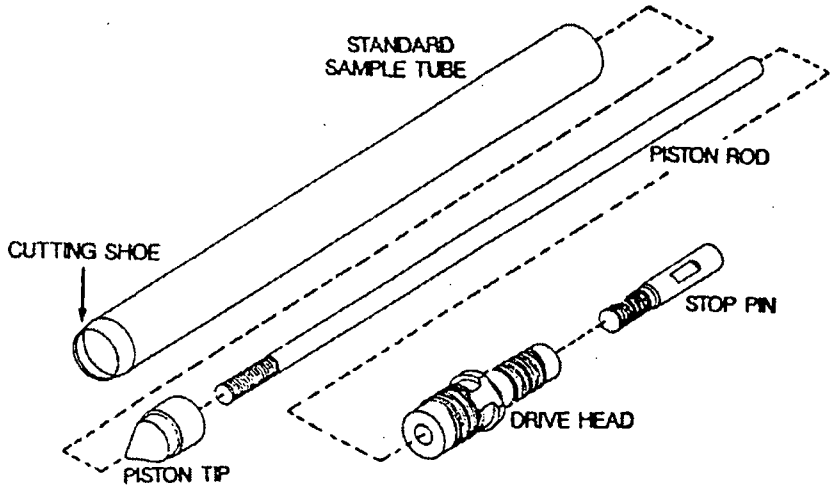


FIGURE 5

PROBE-DRIVE SYSTEM



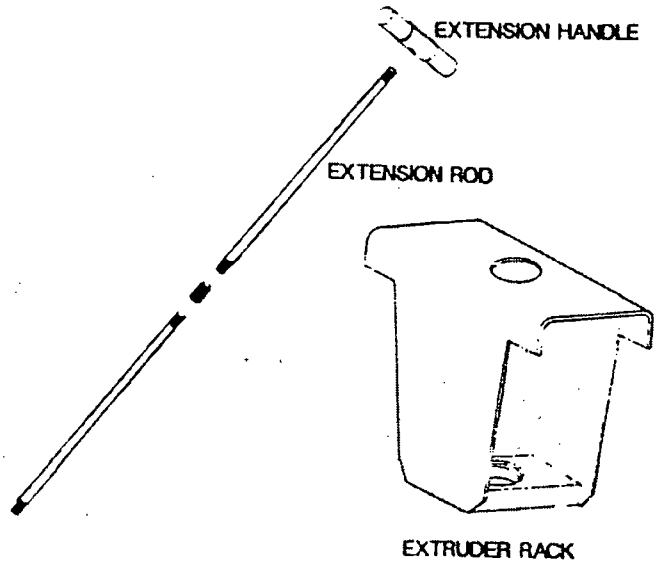
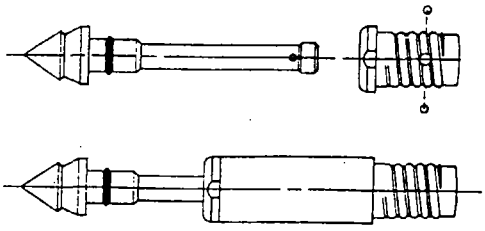
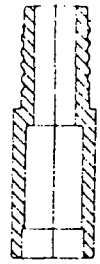


FIGURE 6

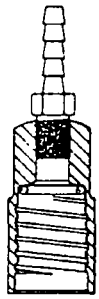
STANDARD SOIL GAS TOOLS



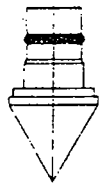
RETRACTABLE POINT HOLDER



EXPENDABLE POINT HOLDER



GAS SAMPLING CAP



EXPENDABLE POINT

FIGURE 7

POST-RUN TUBING (PRT) SYSTEM

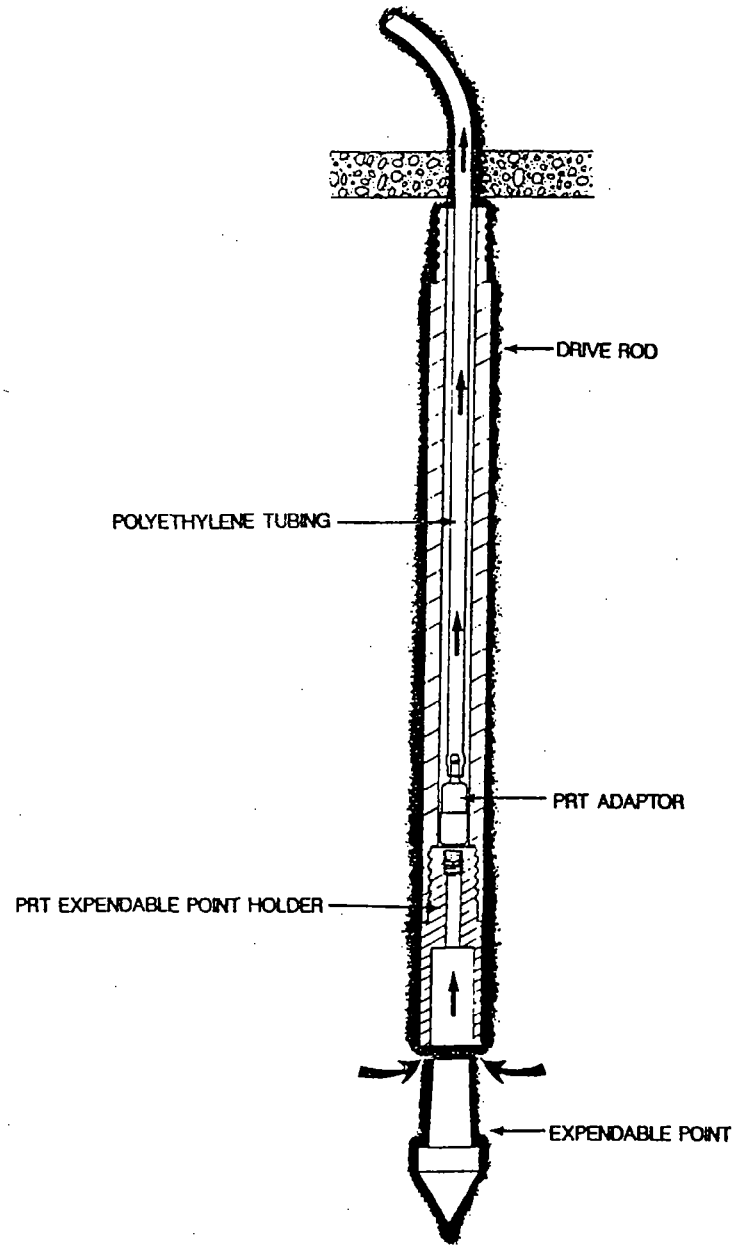
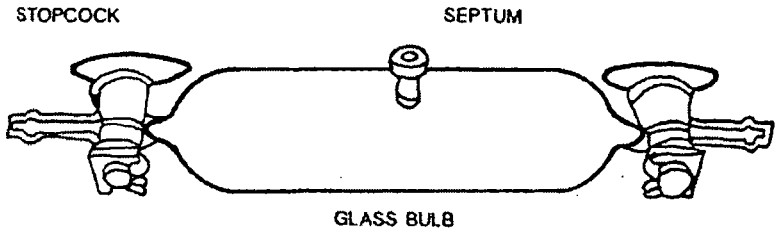


FIGURE 8

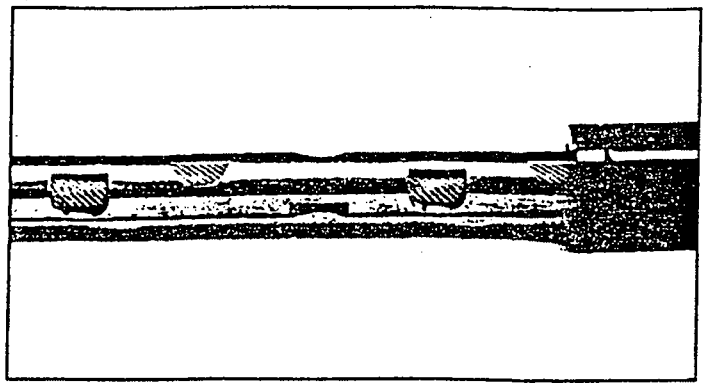
SOIL GAS SAMPLE CONTAINER



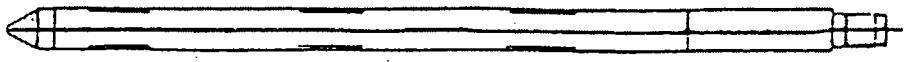
Note: Tedlar bags are also used for collection of soil gas samples; however, they are not shown on this figure.

FIGURE 9

GROUNDWATER SAMPLING TOOLS



SCREEN POINT SAMPLER IN OPEN POSITION



MILL-SLOTTED WELL POINT

FIGURE 10

PIEZOMETER INSTALLATION

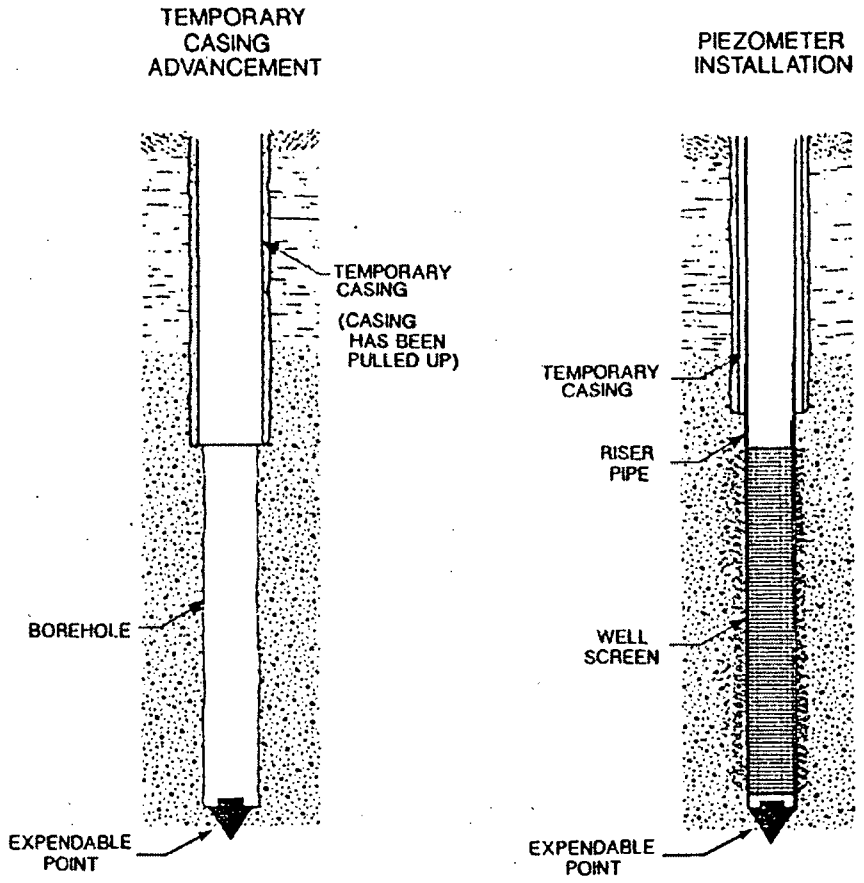
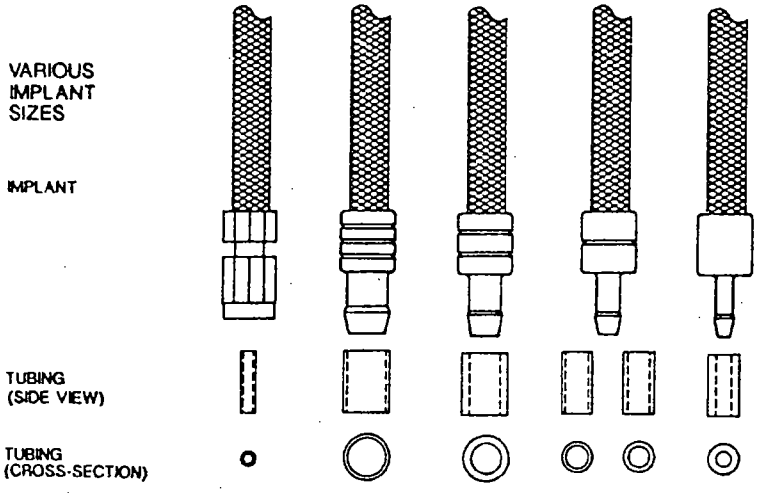
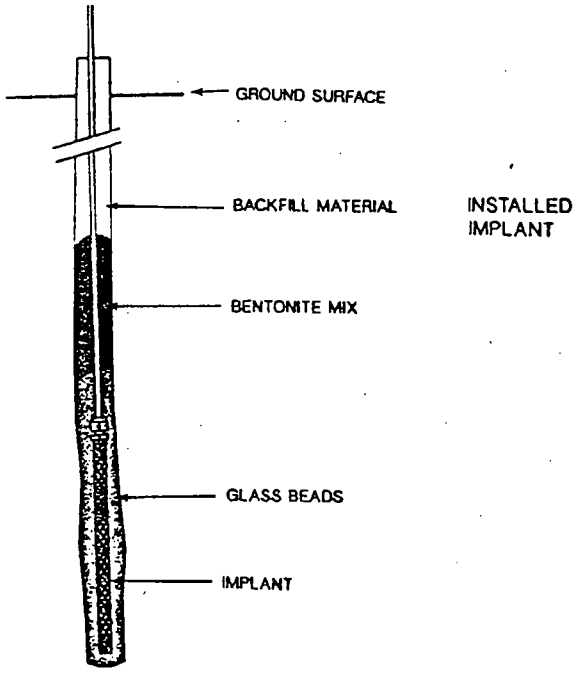


FIGURE 11

VAPOR SAMPLING IMPLANTS





SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SLUDGE AND SEDIMENT SAMPLING

SOP NO. 006

REVISION NO. 3

Last Reviewed: January 2000

Quality Assurance Approved

May 18, 1993

Date

1.0 BACKGROUND

Sludges are semisolid materials ranging from dewatered solids to high-viscosity liquids. Sludges generally accumulate as residuals of water-bearing waste treatment or industrial process systems. Sludges typically accumulate in tanks, drums, impoundments, or other types of containment systems.

Sediments generally are materials deposited in surface impoundments or in natural waterways such as lakes, streams, and rivers.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for sampling sludge in open drums and shallow tanks (3 feet deep or less) and sediment in lakes, streams, and rivers.

1.2 SCOPE

This SOP applies to collection of sludge and sediment samples. It provides detailed procedures for gathering such samples with specific equipment.

1.3 DEFINITIONS

Gravity Corer: Metal tube with a tapered nosepiece on the bottom and a check valve on the top. The nosepiece reduces core disturbance during penetration. The check valve allows air and water to pass through the sampler during deployment and prevents sample loss (washout) during retrieval.

Hand Corer: Thin-wall metal tube with a tapered nosepiece, a "T" handle to facilitate sampler deployment and retrieval, and a check valve on top.

Ponar Grab Sampler: A clamshell-type metal scoop activated by a counter-lever latching system.

1.4 REFERENCES

American Public Health Association. 1975. "Standard Methods for the Examination of Water and Wastewater." 14th Edition. Washington DC.

U.S. Environmental Protection Agency (EPA). 1984. "Characterization of Hazardous Waste Sites -- A Methods Manual. Volume II -- Available Sampling Methods." Second Edition. EPA-600/A-84-076. December.

EPA. 1994. "Sediment Sampling." Environmental Response Team SOP #2016 (Rev. #0.0, 11/17/94). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

The selection of sampling equipment and procedures should be based on project objectives and site-specific conditions such as the type and volume of sludge or sediment to be sampled, sampling depth, and the type of sample required (disturbed or undisturbed). The selected sampling equipment should be constructed of inert materials that will not react with the sludge or sediment being sampled.

The following equipment may be required to sample sludge or sediment:

- Plastic sheeting
- Field logbook
- Spoons or spatulas
- Stainless-steel scoop or trowel
- Gravity corer
- Ponar grab sampler
- Stainless-steel or Teflon® tray
- Hand corer
- Nylon rope
- Sample containers and labels
- Chain-of-custody and shipping materials
- Decontamination materials

2.0 PROCEDURES

This section provides general procedures for sampling sludge and sediment. Sections 2.1 through 2.4 specify the methods and equipment to be used for such sampling.

Sludge Sampling

Sludge can often be sampled using a stainless-steel scoop or trowel (see Section 2.1). Frequently sludge forms when components with higher densities settle out of a liquid. When this happens, the sludge may still have an upper liquid layer above the denser components. When the liquid layer is sufficiently shallow, the sludge may be sampled using a hand corer (see Section 2.2). Use of the hand corer is preferred because it results in less sample disturbance. The hand corer also allows for the collection of an aliquot of the overlying liquid. This prevents drying or excessive oxidation of a sample before analysis. The hand corer may also be adapted to hold a brass, polycarbonate plastic, or Teflon® liner.

A gravity corer may also be used to collect samples of most sludges and sediments (see Section 2.3). A gravity corer is capable of collecting an undisturbed sample that profiles the strata present in a sludge or sediment. Depending on the weight of the gravity corer and the density of the sludge or sediment, a gravity corer may penetrate the material up to 30 inches. If the layer is shallow (less than 1 foot), gravity corer and hand corer penetration may damage any underlying liner or confining layer. In such situations, a Ponar grab sampler may be used because it is generally capable of penetrating only a few inches (see Section 2.4).

Sediment Sampling

Sediment can be sampled in much the same manner as sludge; however, a number of additional factors must be considered. In streams, lakes, and impoundments, for instance, sediment is likely to demonstrate significant variations in composition.

For stream sediment sampling, the sampling location farthest downstream should be sampled first. Sediment samples collected in upstream and downstream locations should be obtained in similar

depositional environments and, whenever possible, should be obtained from slow-moving pools. In addition, a sediment sample should be collected at approximately the same location as an associated aqueous sample. Aqueous samples should be obtained first to avoid collecting suspended particles that may result from sediment sampling. To avoid disturbing an area to be sampled, sampling locations in streams should always be approached from the downstream side.

Sediment samples collected from lakes and impoundments should also be collected at approximately the same locations as associated aqueous samples. As in stream sampling, aqueous samples should be collected first to avoid collecting suspended particles that may result from sediment sampling. Downgradient and background samples should be collected from similar depositional environments.

Exact sampling locations should be documented in field logbooks or on data sheets with respect to fixed reference points. In addition, the presence of rocks, debris, or organic material in the sludge or sediment to be sampled may preclude use or require modification of sampling equipment.

The following subsections specify methods for sludge or sediment sampling with specific equipment.

2.1 SAMPLING WITH A SCOOP OR TROWEL

Sludge or sediment samples may be collected with a simple scoop or trowel. This method is more applicable to sludge but can also be used for sediments, provided that the water is very shallow (a few inches). However, using a scoop or trowel may disrupt the water-sediment interface and cause substantial sample alteration. This method provides a simple, quick means of collecting a disturbed sample of sludge or sediment.

The following procedure can be used for sampling sludge or sediment with a scoop or trowel:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

2. Affix a completed sample container label to the appropriate sample container.

3. Carefully insert a precleaned scoop or trowel into the sludge or sediment and remove the sample. In the case of sludge exposed to air, remove the first 2 to 4 inches of material before collecting the sample.

4. When compositing a series of grab samples, combine the samples in a stainless-steel bowl or Teflon® tray.

5. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

6. If required, preserve the sample in accordance with SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

7. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

8. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements.

9. Decontaminate all nondisposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.2 SAMPLING WITH A HAND CORER

The hand corer (see Figure 1) is used in the same situations and for the same materials as those described for the use of a scoop or trowel (see Section 2.1). However, the hand corer may be used to collect an undisturbed sample that can profile any stratification resulting from changes in material deposition.

Some hand corers can be fitted with extension handles that allow collection of samples underlying a shallow layer of liquid. Most hand corers can be adapted to hold liners, which are generally available in brass, polycarbonate plastic, or Teflon®. A liner material should be chosen that will not compromise the intended analytical procedures.

The following procedure can be used for sampling sludge or sediment with a hand corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

2. Affix a completed sample container label to the appropriate sample container.

3. Position a precleaned hand corer above the sampling location. Carefully deploy the hand corer into the sludge or sediment using a smooth, continuous motion.

4. When the hand corer is at the desired depth, rotate the "T" handle and retrieve the hand corer using a single, smooth motion.

5. Remove the nosepiece and extract the sample. Place the sample on a clean stainless-steel or Teflon® tray.

6. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

7. If required, preserve the sample in accordance with SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

8. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

9. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements.

10. Decontaminate all nondisposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.3 SAMPLING WITH A GRAVITY CORER

A gravity corer (see Figure 2) can collect essentially undisturbed samples to profile strata that develop in sediment and sludge during the deposition process. Depending on the sediment or sludge density and the gravity corer's weight, the sampler typically can penetrate the sediment or sludge to a depth of 30 inches.

Gravity corers should be used carefully in open drums, shallow tanks, or lagoons with liners. A gravity corer could penetrate beyond the sludge or sediment layer and damage the liner material.

The following procedure can be used for sampling sludge or sediment with a gravity corer:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

2. Affix a completed sample container label to the appropriate sample container.

3. Attach the required length of sample line to a precleaned gravity corer. Braided, 3/16-inch nylon line is sufficient; however, 3/4-inch nylon line is easier to grasp during hoisting.

4. Secure the free end of the line to a fixed support to prevent accidental loss of the gravity corer.

5. Position the gravity corer above the sampling location. Allow the gravity corer to fall freely through the liquid and penetrate the sludge or sediment layer.

6. Retrieve the gravity corer with a smooth, continuous lifting motion. Do not bump the corer, as this may result in some sample loss.

7. Remove the nosepiece from the gravity corer. Slide the sample out of the corer into a stainless-steel or Teflon® pan.

8. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

9. If required, preserve the sample in accordance with SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

10. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

11. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements.

12. Decontaminate all nondisposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.4 SAMPLING WITH A PONAR GRAB SAMPLER

A Ponar grab sampler (see Figure 3) can be used to sample most types of sludges and sediments. Its penetration depth usually does not exceed several inches. The Ponar grab sampler, like other grab samplers, cannot collect undisturbed samples; therefore, this sampler should be used only after all overlying water samples have been collected.

The following procedure can be used for sampling sludge or sediment with a Ponar grab sampler:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

2. Affix a completed sample container label to the appropriate sample container.

3. Attach the required length of sample line to a precleaned Ponar grab sampler. Braided, 3/4-inch nylon line is recommended for ease in hoisting.

4. Measure the distance from the water surface or other reference point to the top of the sludge or sediment. Mark this measurement on the sample line. To avoid unnecessary disturbance of the sludge or sediment from lowering the Ponar grab sampler too quickly, it is recommended that a second mark be made on the sample line to indicate the proximity of the reference mark.

5. Open the Ponar sampler's jaws until they are latched. The jaws will be triggered if the Ponar sampler comes in contact with or is supported by anything other than the sample line. Tie the free end of the sample line to a fixed support.

6. Position the Ponar grab sampler above the sampling location. Lower the sampler until the proximity mark is reached. Then, slowly lower the Ponar grab sampler until it touches and penetrates the sludge or sediment.

7. Allow the sample line to slacken a few inches to release the latching mechanism that closes the sampler's jaws. As the jaws close, they scoop the sludge or sediment up into the sampler. More slack may be required when sampling in surface waters with strong currents.

8. Retrieve the sampler and release its contents into a stainless-steel or Teflon® tray.

9. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

10. If required, preserve the sample in accordance with SOP No. 016, Sample Container, Preservation, and Maximum Holding Time Requirements.

11. Ensure that a Teflon® liner is present in the sample container cap, if required. Secure the cap tightly on the sample container.

12. Complete all chain-of-custody documents, field logbook entries, and sample packaging requirements.

13. Decontaminate all nondisposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

FIGURE 1

HAND CORER

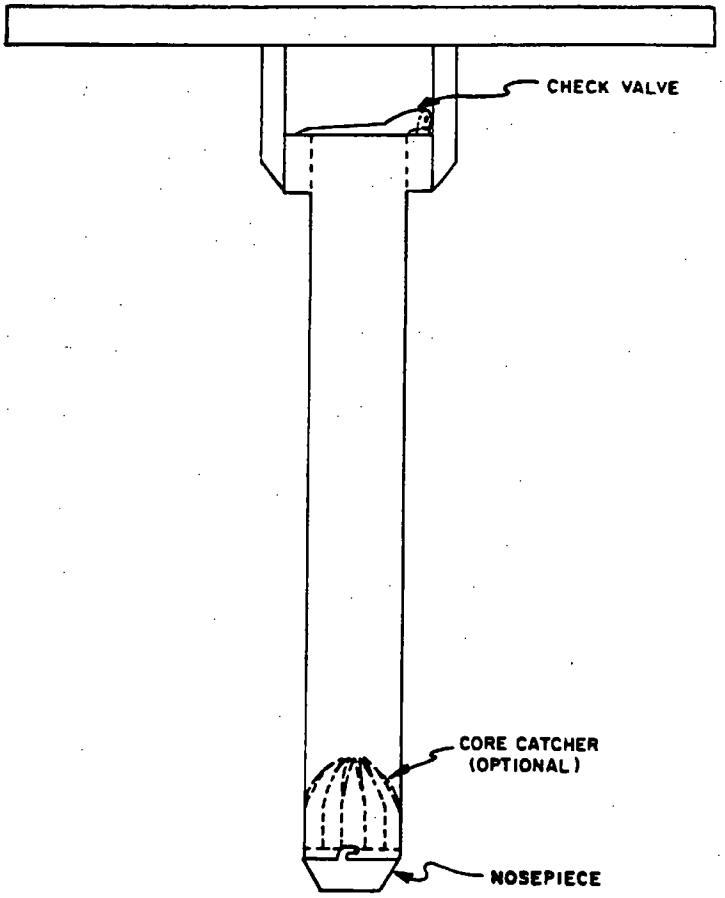


FIGURE 2

GRAVITY CORER

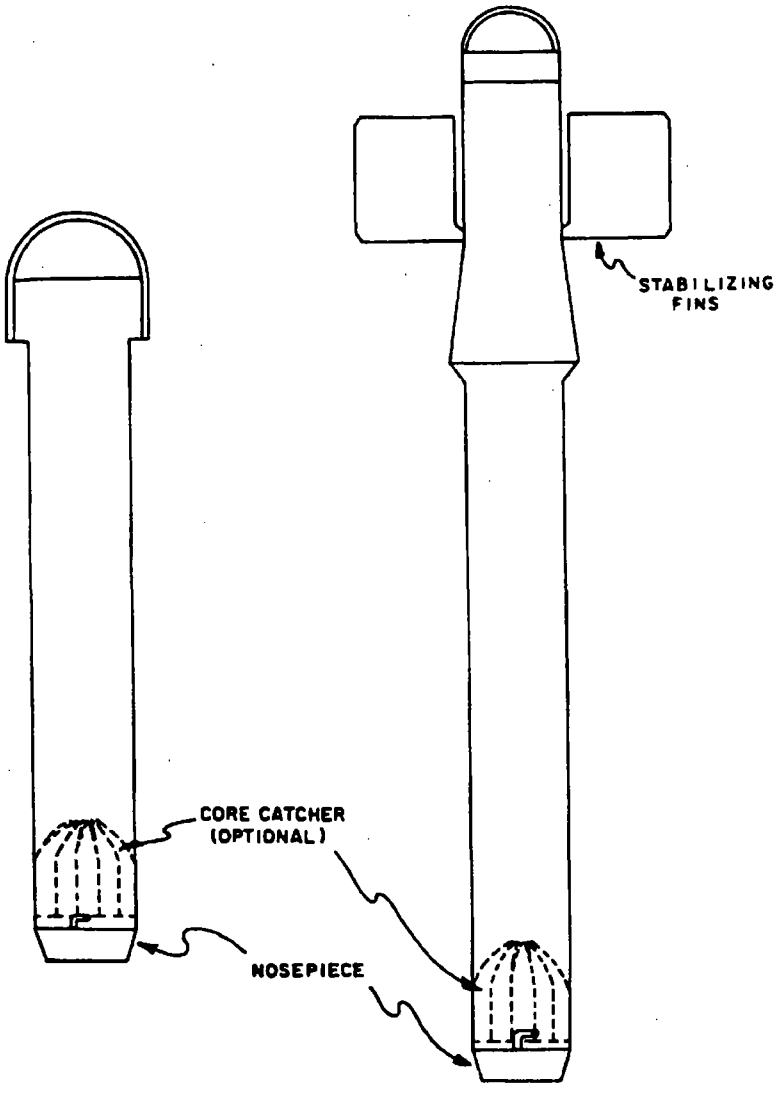
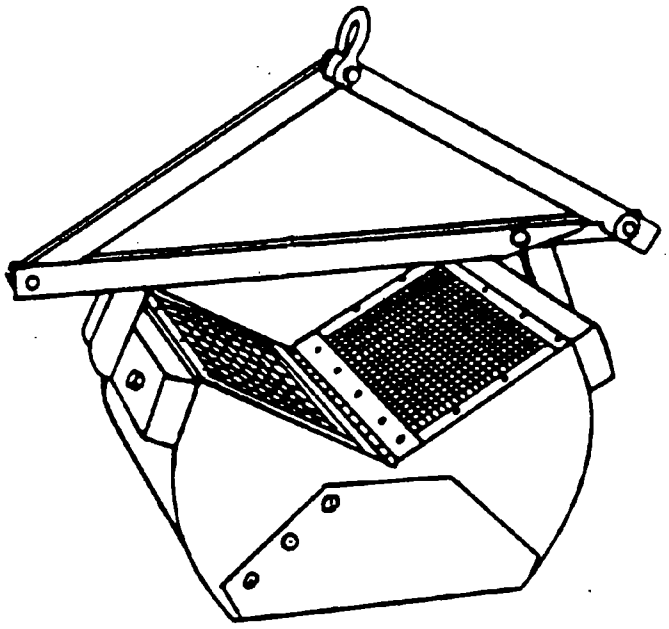


FIGURE 3

PONAR GRAB SAMPLER



SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

BULK MATERIALS SAMPLING

SOP NO. 007

REVISION NO. 2

Last Reviewed: December 1999

Quality Assurance Approved

May 19, 1993

Date

1.0 BACKGROUND

Bulk materials are typically sampled to characterize a homogeneous collection of a single, identifiable product.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for sampling bulk materials.

1.2 SCOPE

This SOP applies to field sampling of bulk materials with a scoop, trier, or grain thief. It provides detailed procedures for gathering such samples with specific sampling equipment.

1.3 DEFINITIONS

HNu® Photoionization Detector (HNu®): A direct-reading air monitoring instrument used to measure the level of organic vapors in air.

Organic Vapor Analyzer (OVA®): An air monitoring instrument used to measure the level of organic vapors in air based on flame ionization.

Grain Thief: A sampling device made of two slotted, concentric, telescoping tubes designed to penetrate solid material.

Trier: A sampling device consisting of a long tube cut in half lengthwise with a sharpened tip that allows the sampler to cut into sticky solids and to loosen cohesive soil.

1.4 REFERENCES

deVera, E.R., and others. 1980. "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA-600/2-80-018. January.

Horwitz, W., and others. 1979. "Animal Feed: Sampling Procedure." *Official Methods of Analysis*. The Association of Official Analytical Chemists. 12th Edition. Washington, DC.

U.S. Environmental Protection Agency. 1984. "Characterization of Hazardous Waste Sites—A Methods Manual: Volume II. Available Sampling Methods." Second Edition. EPA-600/4-84-076. December.

1.5 REQUIREMENTS AND RESOURCES

Sampling of bulk materials can be performed by a variety of equipment. The selection of sampling equipment and procedures should be based on site-specific conditions such as the type and volume of material to be sampled. The selected sampling equipment should be constructed of inert materials that will not react with the material being sampled. The following equipment may be required to sample bulk materials:

- Trier
- Scoop
- Trowel
- Grain thief
- Spoons or spatulas
- OVA® or HNu®
- Decontamination materials
- Sample containers and labels
- Chain-of-custody and shipping materials
- Field logbook
- Stainless-steel or Teflon® tray

Additional resources for sampling bulk materials are discussed in *The Sampling of Bulk Materials* by R. Smith and G. V. James of the Royal Society of Chemistry, London (1981). Although this book does not

deal specifically with hazardous waste sampling, the concepts discussed are applicable, especially those regarding establishment of a sampling scheme.

2.0 PROCEDURES

Bulk materials are usually contained in bags, drums, or hoppers, although large amounts of material may be piled on the ground, either deliberately or as the result of a spill.

Material surfaces exposed to the atmosphere may undergo chemical alteration or degradation and should be removed before initiating sample collection. Because the process conditions that produced the bulk material may have varied over time, a series of samples should be collected and composited into one sample to represent the material. Samples collected for volatile organic compound analysis should not be composited.

The following sections provide detailed procedures for sampling bulk materials with a trier, scoop, or trowel, and with a grain thief.

2.1 SAMPLING BULK MATERIALS WITH A TRIER, SCOOP, OR TROWEL

A typical trier (Figure 1) is a long tube with a slot that extends almost its entire length. The tip and edges of the tube slot are sharpened to allow the trier to cut a core after being inserted into the material. A trier is most useful when sampling moist or sticky solids and powdered or granular material with a particle diameter less than half the diameter of the trier. Sampling triers are usually made of stainless steel and have wooden handles. Triers are 24 to 40 inches long and 0.5 to 1.0 inch in diameter. They can be purchased from laboratory supply companies.

A scoop or trowel may be used for sampling bulk materials as well as dry, granular, or powdered material in bins or other shallow containers. A scoop is preferred over a trowel because the scoop is usually made of materials less subject to corrosion and chemical reactions.

A trowel is shaped like a small shovel; the blade is usually 3 to 5 inches long and has a sharp tip. A scoop is similar to a trowel, but the blade is usually more curved and has a closed upper end to contain the sampled material. Scoops are available in different sizes and shapes; stainless-steel and polypropylene scoops with blades 3 to 6 inches long are recommended. Trowels can be purchased from hardware stores; scoops are generally available from laboratory supply companies.

The following procedure can be used to sample bulk materials with a trier, scoop, or trowel:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017, Sample Collection Container Requirements.

2. Wear appropriate protective clothing and gear. Use an HNu® or OVA® to monitor for levels of volatile organic vapors that may be present in accordance with SOP No. 003, Organic Vapor Air Monitoring.

3. Affix a completed sample container label to the appropriate sample container.

4. Insert a clean trier, scoop, or trowel (implement) into the material at a 0 to 45 degree angle from horizontal. This orientation minimizes sample spillage.

5. If the material is cohesive, rotate the implement once or twice to cut a core of material.

6. Slowly withdraw the implement, making sure the slot or blade is facing upward.

7. If composite sampling is required, repeat steps 4 through 6 at different points two or more times. Combine the samples in a stainless-steel bowl or similar container.

8. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

9. Ensure that a Teflon® liner is present in the cap of the sample container cap, if required. Secure the cap tightly on the sample container.

10. Complete all chain-of-custody documents, field logbook entries, and packaging requirements.

11. Decontaminate all nondisposable sampling implements after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

2.2 SAMPLING BULK MATERIALS WITH A GRAIN THIEF

A grain thief is used for sampling powdered or granular materials in bags, fiber drums, sacks, or similar containers. This sampler is useful when the material contains particles no greater than 0.25 inch in diameter.

A grain thief (Figure 2) consists of two slotted, concentric, telescoping tubes, usually made of brass or stainless steel. The outer tube has a conical, pointed tip on one end that permits the sampler to penetrate the material being sampled. The sampler is about 24 to 40 inches long and 0.5 to 1 inch in diameter. Grain thieves are commercially available from laboratory supply companies.

The following procedure can be used to sample bulk materials with a thief:

1. Place all sampling equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017, Sample Collection Container Requirements.

2. Wear appropriate protective clothing and gear. Use an HNu® or OVA® to monitor for levels of volatile organic vapors that may be present in accordance with SOP No. 003, Organic Vapor Air Monitoring.

3. Affix a completed sample container label to an appropriate sample container.

4. Insert a clean grain thief in the closed position into the material. Insert it from a point near the top edge or corner of the material, through the center, and to a point opposite the point of entry.

5. Rotate the inner tube of the grain thief into the open position. Wiggle the thief a few times to allow the material being sampled to enter the open slots. Close the grain thief and withdraw it from the material.

6. Place the grain thief in a horizontal position with the slots facing upward. Rotate the outer tube and slide it away from the inner tube.

7. If composite sampling is required, repeat steps 4 through 6 at different points two or more times. Combine the samples in a stainless-steel bowl or similar container.

8. Transfer the sample into the labeled container using a stainless-steel or plastic spoon, spatula, or similar tool.

9. Ensure that a Teflon® liner is present in the cap of the sample container, if required. Secure the cap tightly on the sample container.

10. Complete all chain-of-custody documents, field logbook entries, and packaging requirements.

11. Decontaminate all nondisposable sampling equipment after each use and between sampling locations using the procedures in SOP No. 002, General Equipment Decontamination.

FIGURE 1

TRIER

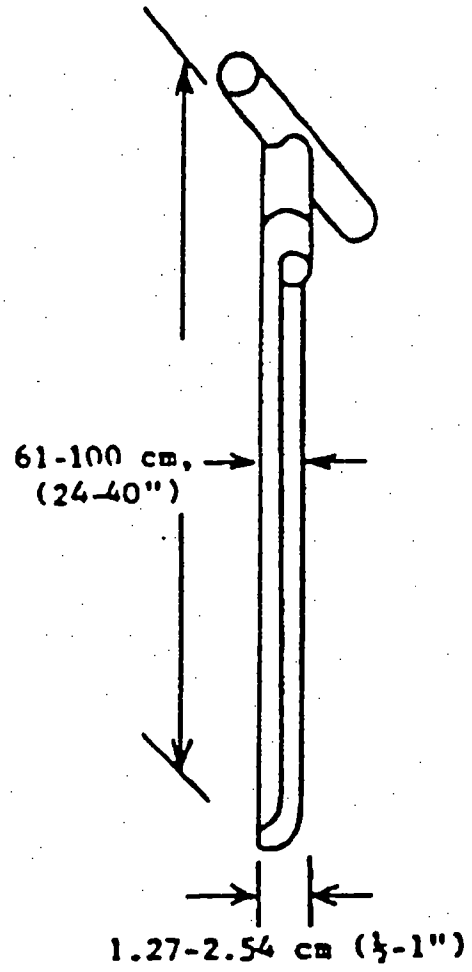
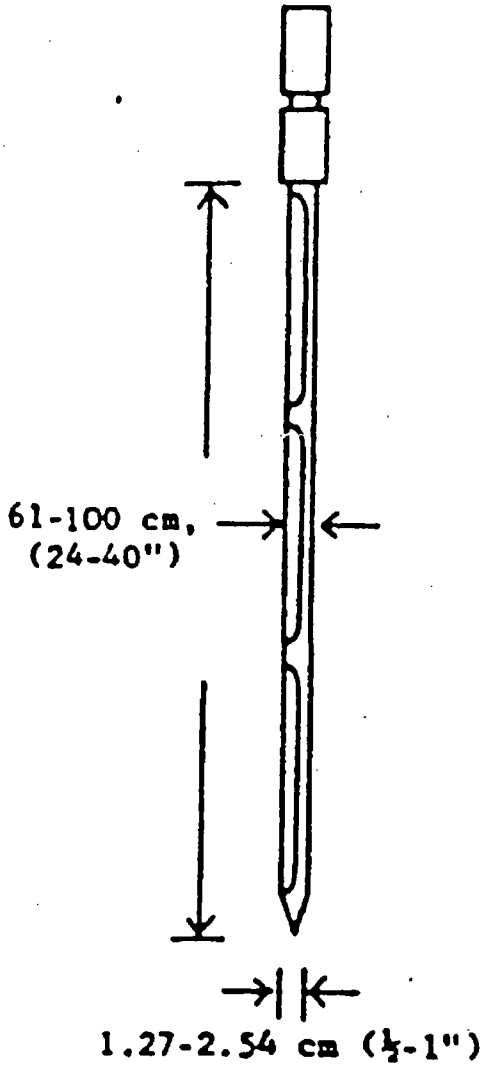


FIGURE 2

GRAIN THIEF



METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As K_β line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD = Relative standard deviation for the precision measurement for the analyte
SD = Standard deviation of the concentration for the analyte
Mean concentration = Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only "screening" type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.

[a] All concentrations in milligrams per kilogram.

%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

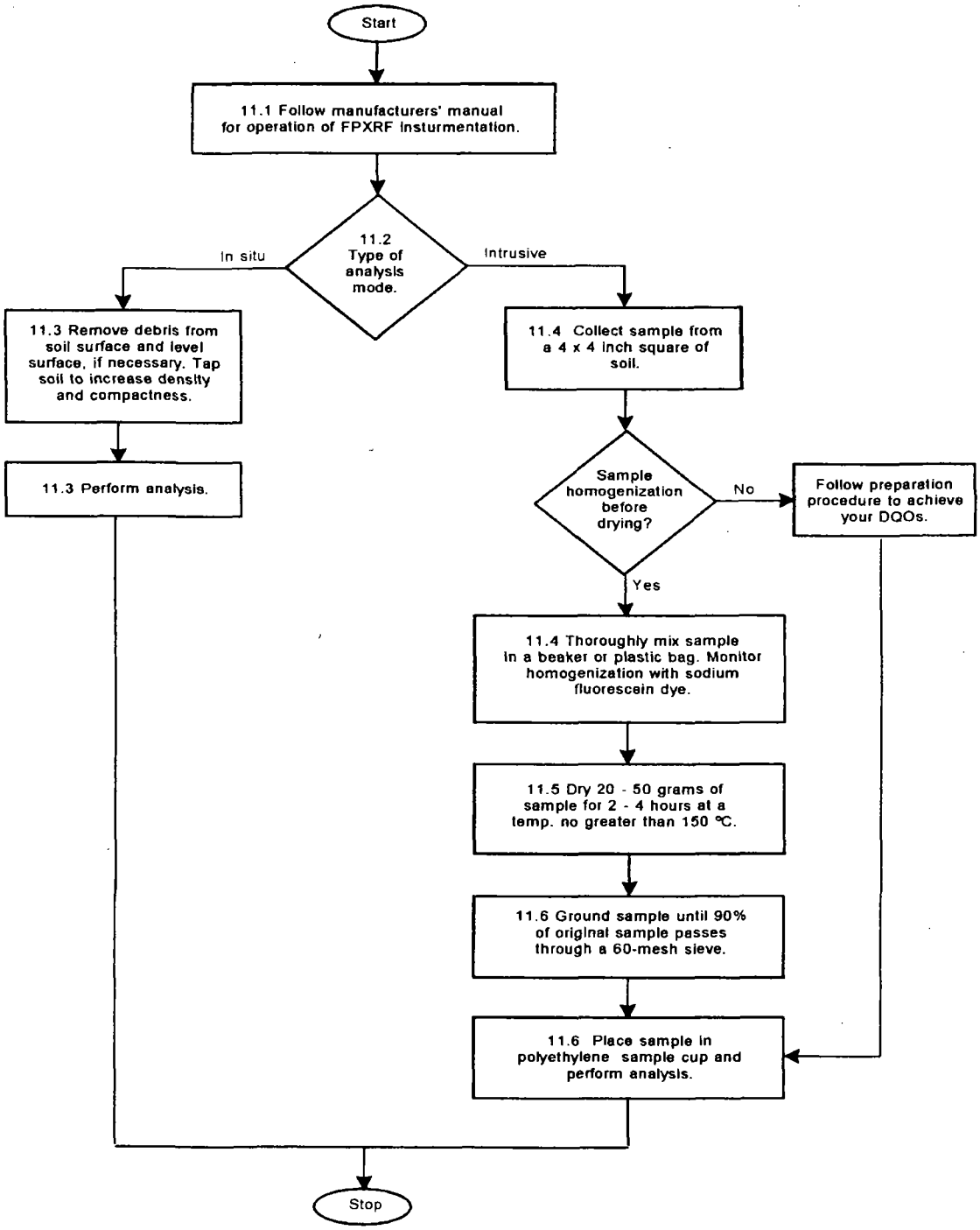
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

MONITORING WELL INSTALLATION

SOP NO. 020

REVISION NO. 3

Last Reviewed: December 2000

Quality Assurance Approved

December 19, 2000

Date

1.0 BACKGROUND

Groundwater monitoring wells are designed and installed for a variety of reasons including: (1) detecting the presence or absence of contaminants, (2) collecting groundwater samples representative of in situ aquifer chemical characteristics, or (3) measuring water levels for determining groundwater potentiometric head and groundwater flow direction.

Although detailed specifications for well installation may vary in response to site-specific conditions, some elements of well installation are common to most situations. This standard operating procedure (SOP) discusses common methods and minimum standards for monitoring well installation for Tetra Tech EM Inc. (Tetra Tech) projects. The SOP is based on widely recognized methods described by the U.S. Environmental Protection Agency (EPA) and American Society for Testing and Materials (ASTM). However, well type, well construction, and well installation methods will vary with drilling method, intended well use, subsurface characteristics, and other site-specific criteria. In addition, monitoring wells should be constructed and installed in a manner consistent with all local and state regulations. Detailed specifications for well installation should be identified within a site-specific work plan, sampling plan, or quality assurance project plan (QAPP).

General specifications and installation procedures for the following monitoring well components are included in this SOP:

- Monitoring well materials
  - Casing materials
  - Well screen materials
  - Filter pack materials
  - Annular sealant (bentonite pellets or chips)
  - Grouting materials
  - Tremie pipe
  - Surface completion and protective casing materials
  - Concrete surface pad and bumper posts
  - Uncontaminated water
- Monitoring well installation procedures
  - Well screen and riser placement
  - Filter pack placement
  - Temporary casing retrieval

  - Annular seal placement
  - Grouting
  - Surface completion and protective casing (aboveground and flush-mount)
  - Concrete surface pad and bumper posts
  - Permanent and multiple casing well installation

- Recordkeeping procedures
  - Surveying
  - Permits and well construction records
  - Monitoring well identification

Well installation methods will depend to some extent on the boring method. Specific boring or drilling protocols are detailed in other SOPs. The boring method, in turn, will depend on site-specific geology and hydrogeology and project requirements. Boring methods commonly used for well installation include:

- Hollow-stem augering
- Cable tool drilling
- Mud rotary drilling
- Air rotary drilling
- Rock coring

The hollow-stem auger method is preferred in areas where subsurface materials are unconsolidated or loosely consolidated and where the depth of the boring will be less than 100 feet. This maximum effective depth for hollow-stem augering depends on the diameter of the augers, the formation characteristics, and the strength and durability of the drilling equipment. This method is preferred because under the right conditions it is cost effective, addition of water into the subsurface is limited, continuous soil samples can easily be collected, and monitoring wells can easily be constructed within the hollow augers.

Cable tool drilling is a preferred method when the subsurface contains boulders, coarse gravels, or flowing sands, or when the operational depth of the hollow-stem auger is exceeded. However, this method is slow.

Rotary methods are generally used when other methods cannot be used. The use of drilling fluids or large amounts of water to maintain an open borehole, and the difficulty in obtaining representative samples limit the utility of rotary methods. However, rotary methods can be used to quickly and effectively drill deep wells through consolidated or unconsolidated materials. Modifications to this method such as dual-tube

drilling procedures, drill-through casing hammers, or eccentric-type drill systems, can reduce the amount of fluids introduced into the well borehole.

Rock coring is an effective method when drilling in competent consolidated rock. Intact, continuous cores can be obtained, and limited amounts of fluid are required if the formations are not fractured.

1.1 PURPOSE

This SOP establishes the requirements and procedures for monitoring well installation. Monitoring wells should be designed to function properly throughout the duration of the monitoring program. The performance objectives for monitoring well installation are as follows:

- Ensure that the monitoring well will provide water samples representative of in situ aquifer conditions.
- Ensure that the monitoring well construction will last for duration of the project.
- Ensure that the monitoring well will not serve as a conduit for vertical migration of contaminants, particularly vertical migration between discrete aquifers.
- Ensure that the well diameter is adequate for all anticipated downhole monitoring and sampling equipment.

1.2 SCOPE

This SOP applies to the installation of monitoring wells. Although some of the procedures may apply to the installation of water supply wells, this SOP is not intended to cover the design and construction of such wells. The SOP identifies several well drilling methods related to monitoring well installation, but the scope of this SOP does not include drilling methods.

Other relevant SOPs include SOP 002 for decontamination of drilling and well installation equipment, SOP 005 for soil sampling, SOP 021 for monitoring well development, SOPs 010 and 015 for groundwater sampling from monitoring wells, and SOP 014 for measuring static water levels within monitoring wells.

1.3 DEFINITIONS

Annulus: The space between the monitoring well casing and the wall of the well boring.

Bentonite seal: A colloidal clay seal separating the sand pack from the annular grout seal.

Centralizer: A stainless-steel or plastic spacer that keeps the well screen and casing centered in the borehole.

Filter pack: A clean, uniform sand or gravel placed between the borehole wall and the well screen to prevent formation material from entering the screen.

Grout seal: A fluid mixture of (1) bentonite and water, (2) cement, bentonite, and water, or (3) cement and water placed above the bentonite seal between the casing and the borehole wall to secure the casing in place and keep water from entering the borehole.

Tremie pipe: A rigid pipe used to place the well filter pack, bentonite seal, or grout seal. The tremie pipe is lowered to the bottom of the well or area to be filled and pulled up ahead of the material being placed.

Well casing: A solid piece of pipe, typically polyvinyl chloride (PVC) or stainless steel, used to keep a well open in either unconsolidated material or unstable rock.

Well screen: A PVC or stainless steel pipe with openings of a uniform width, orientation, and spacing used to keep materials other than water from entering the well and to stabilize the surrounding formation.

1.4 REFERENCES

American Society for Testing and Materials. 1995. Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers. D5092-90. West Conshohocken, Pennsylvania.

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EPA. 1994. Monitor Well Installation. Environmental Response Team SOP #2048 (Rev. #0.0, 03/18/96). On-Line Address: http://www.ert.org/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

Well installation requires a completed boring with stable or supported walls. The type of drilling rig needed to complete the boring and the well construction materials required for monitoring well installation will depend on the drilling method used, the geologic formations present, and chemicals of concern in groundwater. The rig and support equipment used to drill the borehole is usually used to install the well. Under most conditions, the following items are also required for the proper installation of monitoring wells:

- Tremie pipe and funnel
- Bentonite pellets or chips
- Grouting supplies
- Casing materials
- Well screen materials
- Filter pack materials
- Surface completion materials (protective casing, lockable and watertight well cover, padlock)
- Electronic water level sounding device for water level measurement
- Measuring tape with weight for measuring the depth of the well and determining the placement of filter pack materials
- Decontamination equipment and supplies

- Site-specific work plan, field sampling plan, health and safety plan, and QAPP
- Monitoring Well Completion Record (see Figure 1)

2.0 MONITORING WELL INSTALLATION PROCEDURES

This section presents standard procedures for monitoring well installation and is divided into three subsections. Section 2.1 addresses monitoring well construction materials, while Section 2.2 describes typical monitoring well installation procedures. Section 2.3 addresses recordkeeping requirements associated with monitoring well installation. Monitoring well installation procedures described in work plans, sampling plans, and QAPPs should be fully consistent with the procedures outlined in this SOP as well as any applicable local and state regulations and guidelines.

2.1 MONITORING WELL CONSTRUCTION MATERIALS

Monitoring well construction materials should be specified in the site-specific work plan as well as in the statement of work for any subcontractors assisting in the well installation. Well construction materials that come in contact with groundwater should not measurably alter the chemical quality of groundwater samples with regard to the constituents being examined. The riser, well screen, and filter pack and annular sealant placement equipment should be steam cleaned or high-pressure water cleaned immediately prior to well installation. Alternatively, these materials can be certified by the manufacturer as clean and delivered to the site in protective wrapping. Samples of the filter pack, annular seal, and mixed grout should be retained as a quality control measure until at least one round of groundwater sampling and analysis is completed.

This section discusses material specifications for the following well construction components: casing, well screen, filter pack, annular sealant (bentonite pellets or chips), grout, tremie pipes, surface completion components (protective casing, lockable and water tight cap, and padlock), concrete surface pad, and uncontaminated water. Figure 2 shows the construction details of a typical monitoring well.

2.1.1 Casing Materials

The material type and minimum wall thickness of the casing should be adequate to withstand the forces of installation. If the casing has not been certified as clean by the manufacturer or delivered to and maintained in clean condition at the site, the casing should be steam cleaned or high-pressure water cleaned with water from a source of known chemistry immediately prior to installation (see Tetra Tech SOP No. 002). The ends of each casing section should be either flush-threaded or beveled for welding.

Schedule 40 or Schedule 80 PVC casing is typically used for monitoring well installation. Either type of casing is appropriate for monitoring wells with depths less than 100 feet below ground surface (bgs). If the well is deeper than 100 feet bgs, Schedule 80 PVC should be used.

Stainless steel used for well casing is typically Type 304 and is of 11-gauge thickness.

2.1.2 Well Screen Materials

Well screens should be new, machine-slotted or continuous wrapped wire-wound, and composed of materials most suited for the monitoring environment based on site characterization findings. Well screens are generally constructed of the same materials used for well casing (PVC or stainless steel). The screen should be plugged at the bottom with the same material as the well screen. Alternatively, a short (1- to 2-foot) section of casing material with a bottom (sump) should be attached below the screen. This assembly must be able to withstand installation and development stresses without becoming dislodged or damaged. The length of the slotted area should reflect the interval to be monitored.

If the well screen has not been certified as clean by the manufacturer or delivered to and maintained in clean condition at the site, the screen should be steam cleaned or high-pressure water cleaned with water from a source of known chemistry immediately prior to installation (see Tetra Tech SOP No. 002).

The minimum internal diameter of the well screen should be chosen based on the particular application. A minimum diameter of 2 inches is usually needed to allow for the introduction and withdrawal of sampling devices. Typical monitoring well screen diameters are 2 inches and 4 inches.

The slot size of the well screen should be determined relative to (1) the grain size of particles in the aquifer to be monitored and (2) the gradation of the filter pack material.

Screen length and monitoring well diameter will depend on site-specific considerations such as intended well use, contaminants of concern, and hydrogeology. Some specific considerations include the following:

- Water table wells should have screens of sufficient length and diameter to monitor the water table and provide sufficient sample volume under high and low water table conditions.
- Wells with low recharge should have screens of sufficient length and diameter so that adequate sample volume can be collected.
- Wells should be screened over sufficiently short intervals to allow for monitoring of discrete migration pathways.
- Where light nonaqueous-phase liquids (LNAPL) or contaminants in the upper portion of a hydraulic unit are being monitored, the screen should be set so that the upper portion of the water-bearing zone is below the top of the screen.
- Where dense nonaqueous-phase liquids (DNAPL) are being monitored, the screen should be set within the lower portion of the water-bearing zone, just above a relatively impermeable lithologic unit.
- The screened interval should not extend across an aquiclude or aquitard.
- If contamination is known to be concentrated within a portion of a saturated zone, the screen should be constructed in a manner that minimizes the potential for cross-contamination within the aquifer.
- If downhole geophysical surveys are to be conducted, the casing and screen must be of sufficient diameter and constructed of the appropriate material to allow for effective use of the geophysical survey tools.
- If aquifer tests are to be conducted in a monitoring well, the slot size must allow sufficient flux to produce the required drawdown and recovery. The diameter of the well must be sufficient to house the pump and monitoring equipment, and allow sufficient water flux (in combination with the screen slot size) to produce the required drawdown or recovery.

2.1.3 Filter Pack Materials

The primary filter pack consists of a granular material of known chemistry and selected grain size and gradation. The filter pack is installed in the annulus between the well screen and the borehole wall. The grain size and gradation of the filter pack are selected to stabilize the hydrologic unit adjacent to the screen and to prevent formation material from entering the well during development. After development, a properly filtered monitoring well is relatively free of turbidity.

A secondary filter pack is a layer of material placed in the annulus directly above the primary filter pack and separates the filter pack from the annular sealant. The secondary filter pack should be uniformly graded fine sand, with 100 percent by weight passing through a No. 30 U.S. Standard sieve, and less than 2 percent by weight passing through a No. 200 U.S. Standard sieve.

2.1.4 Annular Sealant (Bentonite Pellets or Chips)

The materials used to seal the annulus may be prepared as a slurry or used as dry pellets, granules, or chips. Sealants should be compatible with ambient geologic, hydrogeologic, and climatic conditions and any man-induced conditions anticipated to occur during the life of the well.

Bentonite (sodium montmorillonite) is the most commonly used annular sealant and is furnished in sacks or buckets in powder, granular, pelletized, or chip form. Bentonite should be obtained from a commercial source and should be free of impurities that may adversely impact the water quality in the well. Pellets are compressed bentonite powder in roughly spherical or disk shapes. Chips are large, coarse, irregularly shaped units of bentonite. The diameter of the pellets or chips should be less than one-fifth the width of the annular space into which they will be placed in order to reduce the potential for bridging. Granules consist of coarse particles of unaltered bentonite, typically smaller than 0.2 inch in diameter. Bentonite slurry is prepared by mixing powdered or granular bentonite with water from a source of known chemistry.

2.1.5 Grouting Materials

The grout backfill that is placed above the bentonite annular seal is ordinarily liquid slurry consisting of either (1) a bentonite (powder, granules, or both) base and water, (2) a bentonite and Portland cement base and water, or (3) a Portland cement base and water. Often, bentonite-based grouts are used when flexibility is desired during the life of the well installation (for example, to accommodate freeze-thaw cycles). Cement- or bentonite-based grouts are often used when cracks in the surrounding geologic material must be filled or when adherence to rock units, or a rigid setting is desired.

Each type of grout mixture has slightly different characteristics that may be appropriate under various physical and chemical conditions. However, quick-setting cements containing additives are not recommended for use in monitoring well installation because additives may leach from the cement and influence the chemistry of water samples collected from the well.

2.1.6 Tremie Pipe

A tremie pipe is used to place the filter pack, annular sealant, and grouting materials into the borehole. The tremie pipe should be rigid, have a minimum internal diameter of 1.0 inch, and be made of PVC or steel. The length of the tremie pipe should be sufficient to extend to the full depth of the monitoring well.

2.1.7 Surface Completion and Protective Casing Materials

Protective casings that extend above the ground surface should be made of aluminum, steel, stainless steel, cast iron, or a structural plastic. The protective casing should have a lid with a locking device to prevent vandalism. Sufficient clearance, usually 6 inches, should be maintained between the top of the riser and the top of protective casing. A water-tight well cap should be placed on the top of the riser to seal the well from surface water infiltration in the event of a flood. A weep hole should be drilled in the casing a minimum of 6 inches above the ground surface to enable water to drain out of the annular space.

Flush-mounted monitoring wells (wells that do not extend above ground surface) require a water-tight protective cover of sufficient strength to withstand heavy traffic. The well riser should be fitted with a locking water-tight cap.

2.1.8 Concrete Surface Pad and Bumper Posts

A concrete surface pad should be installed around each well when the outer protective casing is installed. The surface pad should be formed around the well casing. Concrete should be placed into the formed pad and into the borehole (on top of the grout), typically to a depth of 1 to 3 feet bgs (depending on state, federal, and local regulations). The protective casing is then installed into the concrete. As a general guideline, if the well casing is 2 inches in diameter, the concrete pad should be 3 feet square and 4 inches thick. If the well casing is 4 inches in diameter, the pad should be 4 feet square and 6 inches thick. Round concrete pads are also acceptable.

The finished pad should be sloped so that drainage flows away from the protective casing and off the pad. The finished pad should extend at least 1 inch below grade. If the monitoring wells are located in high traffic areas, a minimum of three bumper posts should be installed around the pad to protect the well. Bumper posts, consisting of steel pipes 3 to 4 inches in diameter and at least 5 feet long, should be installed in a radial pattern around the protective casing, beyond the edges of the cement pad. The base of the bumper posts should be installed 2 feet bgs in a concrete footing; the top of the post should be capped or filled with concrete.

2.1.9 Uncontaminated Water

Water used in the drilling process, to prepare grout mixtures, and to decontaminate the well screen, riser, and annular sealant injection equipment, should be obtained from a source of known chemistry. The water should not contain constituents that could compromise the integrity of the monitoring well installation.

2.2 MONITORING WELL INSTALLATION PROCEDURES

This section describes the procedures used to install a single-cased monitoring well, with either temporary casing or hollow-stem augers to support the walls of the boring in unconsolidated formations. The procedures are described in the order in which they are conducted, and include: (1) placement of well screen and riser pipe, (2) placement of filter pack, (3) progressive retrieval of temporary casing, (4) placement of annular seal, (5) grouting, (6) surface completion and installation of protective casing, and (7) installation of concrete pad and bumper posts.

The additional steps necessary to install a well with permanent or multiple casing strings are described at the end of this section.

2.2.1 Well Screen and Riser Placement

After the total depth of the boring is confirmed and the well screen depth interval and the height of the aboveground completion are determined, the screen and riser is assembled from the bottom up as it is lowered down the hole. The following procedures should be followed:

1. Measure the total depth of the boring using a weighted tape.
2. Determine the length of screen and casing materials required to construct the well.
3. Assemble the well parts from the bottom up, starting with the well sump or cap, well screen, and then riser pipe. Progressively lower the assembled length of pipe.
4. The length of the assembled pipe should not extend above the top of the installation rig.

The well sump or cap, well screen, and riser should be certified clean by the manufacturer or should be decontaminated before assembly and installation. No grease, oil, or other contaminants should contact any portion of the assembly. Flush joints should be tightened, and welds should be water tight and of good quality. The riser should extend above grade and be capped temporarily to prevent entrance of foreign materials during the remaining well completion procedures.

When the well screen and riser assembly is lowered to the predetermined level, it may float and require a method to hold it in place. For borings drilled using cable tool or air rotary drilling methods, centralizers should be attached to the riser at intervals of between 20 and 40 feet.

2.2.2 Filter Pack Placement

The filter pack is placed after the well screen and riser assembly has been lowered into the borehole. The steps below should be followed:

1. Determine the volume of the annular space in the filter pack interval. The filter pack should extend from the bottom of the borehole to at least 2 feet above the top of the well screen.

2. Assemble the required material (sand pack and tremie pipe).

3. Lower a clean or decontaminated tremie pipe down the annulus to within 1 foot of the base of the hole.

4. Pour the sand down the tremie pipe using a funnel; pour only the quantity estimated to fill the first foot.

5. Check the depth of sand in the hole using a weighted tape.

6. Pull the drill casing up ahead of the sand to keep the sand from bridging.

7. Continue with this process (steps 4 through 6) until the filter pack is at the appropriate depth.

If bridging of the filter pack occurs, break out the bridge prior to adding additional filter pack material. For wells less than 30 feet deep installed inside hollow-stem augers, the sand may be poured in 1-foot lifts without a tremie pipe.

Sufficient measurements of the depth to the filter pack material and the depth of the bottom of the temporary casing should be made to ensure that the casing bottom is always above the filter pack. The filter pack should extend 2 feet above the well screen (or more if required by state or local regulations). However, the filter pack should not extend across separate hydrogeologic units. The final depth interval, volume, and type of filter pack should be recorded on the Monitoring Well Completion Record (Figure 1).

A secondary filter pack may be installed above the primary filter pack to prevent the intrusion of the bentonite grout seal into the primary filter pack. A measured volume of secondary filter material should be added to extend 1 to 2 feet above the primary filter pack. As with the primary filter pack, a secondary filter pack must not extend into an overlying hydrologic unit. An on-site geologist should evaluate the need for a secondary filter pack by considering the gradation of the primary filter pack, the hydraulic head difference between adjacent units, and the potential for grout intrusion into the primary filter pack.

The secondary filter material is poured into the annular space through tremie pipe as described above. Water from a source of known chemistry may be added to help place the filter pack into its proper location.

The tremie pipe or a weighed line inserted through the tremie pipe can be used to measure the top of the secondary filter pack as work progresses. The amount and type of secondary filter pack used should be recorded on the Monitoring Well Completion Record (Figure 1).

2.2.3 Temporary Casing Retrieval

The temporary casing or hollow-stem auger should be withdrawn in increments. Care should be taken to minimize lifting the well screen and riser assembly during withdrawal of the temporary casing or auger. It may be necessary to place the top head of the rig on the riser to hold it down. To limit borehole collapse in formations consisting of unconsolidated materials, the temporary casing or hollow-stem auger is usually withdrawn until the lowest point of the casing or auger is at least 2 feet, but no more than 5 feet, above the filter pack. When the geologic formation consists of consolidated materials, the lowest point of the casing or auger should be at least 5 feet, but no more than 10 feet, above the filter pack. In highly unstable formations, withdrawal intervals may be much less. After each increment, the depth to the primary filter pack should be measured to check that the borehole has not collapsed or that bridging has not occurred.

2.2.4 Annular Seal Placement

A bentonite pellet, chip, or slurry seal should be placed between the borehole and the riser on top of the primary or secondary filter pack. This seal retards the movement of grout into the filter pack. The thickness of the bentonite seal will depend on state and local regulations, but the seal should generally be between 3 and 5 feet thick.

The bentonite seal should be installed using a tremie pipe, lowered to the top of the filter pack and slowly raised as the bentonite pellets or slurry fill the space. Care must be taken so that bentonite pellets or chips do not bridge in the augers or tremie pipe.. The depth of the seal should be checked with a weighted tape or the tremie pipe.

If a bentonite pellet or chip seal is installed above the water level, water from a known source should be added to allow proper hydration of the bentonite. Sufficient time should be allowed for the bentonite seal to hydrate. The volume and thickness of the bentonite seal should be recorded on the Monitoring Well Completion Record (Figure 1).

2.2.5 Grouting

Grouting procedures vary with the type of well design. The volume of grout needed to backfill the remaining annular space should be calculated and recorded on the Monitoring Well Completion Record (Figure 1). The use of alternate grout materials, including grouts containing gravel, may be necessary to control zones of high grout loss. Bentonite grouts should not be used in arid regions because of their propensity to desiccate. Typical grout mixtures include the following:

- Bentonite grout: about 1 to 1.25 pounds of bentonite mixed with 1 gallon of water
- Cement-bentonite grout: about 5 pounds of bentonite and one 94-pound bag of cement mixed with 7 to 8 gallons of water
- Cement grout: one 94-pound bag of cement mixed with 6 to 7 gallons of water

The grout should be installed by gravity feed through a tremie pipe. The grout should be mixed in batches in accordance with the appropriate requirements and then pumped into the annular space until full-strength grout flows out at the ground surface without evidence of drill cuttings or fluid. The tremie pipe should then be removed to allow the grout to cure.

The riser should not be disturbed until the grout sets and cures for the amount of time necessary to prevent a break in the seal between the grout and riser. For bentonite grouts, curing times are typically around 24 hours; curing times for cement grouts are typically 48 to 72 hours. However, the curing time required will vary with grout content and climatic conditions. The curing time should be documented in the Monitoring Well Completion Record (Figure 1).

2.2.6 Surface Completion and Protective Casing

Aboveground completion of the monitoring well should begin once the grout has set (no sooner than 24 hours after the grout was placed). The protective casing is lowered over the riser and set into the cured grout. The protective casing should extend below the ground surface to a depth below the frost line (typically 3 to 5 feet, depending on local conditions). The protective casing is then cemented in place. A minimum of 6 inches of clearance should be maintained between the top of the riser and the protective casing. A 0.5-inch diameter drainage or weep hole should be drilled in the protective casing approximately

6 inches above the ground surface to enable water to drain out of the annular space between the casing and riser. A water-tight cap should be placed on top of the riser to seal the well from surface water infiltration in the event of a flood. A lock should be placed on the protective casing to prevent vandalism.

For flush-mounted monitoring wells, the well cover should be raised above grade and the surrounding concrete pad sloped so that water drains away from the cover. The flush-mount completion should be installed in accordance with applicable state and local regulations.

2.2.7 Concrete Surface Pad and Bumper Posts

The concrete pad installed around the monitoring well should be sloped so that the drainage will flow away from the protective casing and off the pad. The finished pad should extend at least 1 inch below grade. If the monitoring wells are located in high traffic areas, a minimum of three bumper posts should be installed in a radial pattern around the protective casing, outside the cement pad. Specifications for concrete surface pads and bumper posts are described in Section 2.1.8.

2.2.8 Permanent and Multiple Casing Well Installation

When wells are installed through multiple saturated zones, special well construction methods should be used to assure well integrity and limit the potential for cross-contamination between geologic zones. Generally, these types of wells are necessary if relatively impermeable layers separate hydraulic units. Two procedures that may be used are described below.

In the first procedure, the borehole is advanced to the base of the first saturated zone. Casing is then anchored in the underlying impermeable layer (aquitard) by advancing the casing at least 1 foot into the aquitard and grouting to the surface. After the grout has cured, a smaller diameter borehole is drilled through the grout. This procedure is repeated until the zone of interest is reached. After the zone is reached, a conventional well screen and riser are set. A typical well constructed in this manner is shown on Figure 3.

A second acceptable procedure involves driving a casing through several saturated layers

while drilling ahead of the casing. However, this method is not acceptable when the driven casing may structurally damage a competent aquitard or aquiclude and result in cross-contamination of the two saturated layers. This method should also be avoided when highly contaminated groundwater or nonaqueous-phase contamination may be dragged down into underlying uncontaminated hydrologic units.

2.3 RECORDKEEPING PROCEDURES

Recordkeeping procedures associated with monitoring well installation are described in the following sections. These include procedures for surveying, obtaining permits, completing well construction records, and identifying monitoring wells.

2.3.1 Surveying

Latitude, longitude, and elevation at the top of the riser should be determined for each monitoring well. A permanent notch or black mark should be made on the north side of the riser. The top of the riser and ground surface should be surveyed.

2.3.2 Permits and Well Construction Records

Local and state regulations should be reviewed prior to monitoring well installation, and any required well permits should be in-hand before the driller is scheduled.

Monitoring well installation activities should be documented in both the field logbook and on the Monitoring Well Completion Record (Figure 1). Geologic logs should be completed and, if necessary, filed with the appropriate regulatory agency within the appropriate time frame.

2.3.3 Monitoring Well Identification

Each monitoring well should have an individual well identification number or name. The well identification may be stamped in the metal surface upon completion or permanently marked by using another method. Current state and local regulations should be checked for identification requirements (such as township, range, section, or other identifiers in the well name).

FIGURE 1

MONITORING WELL COMPLETION RECORD

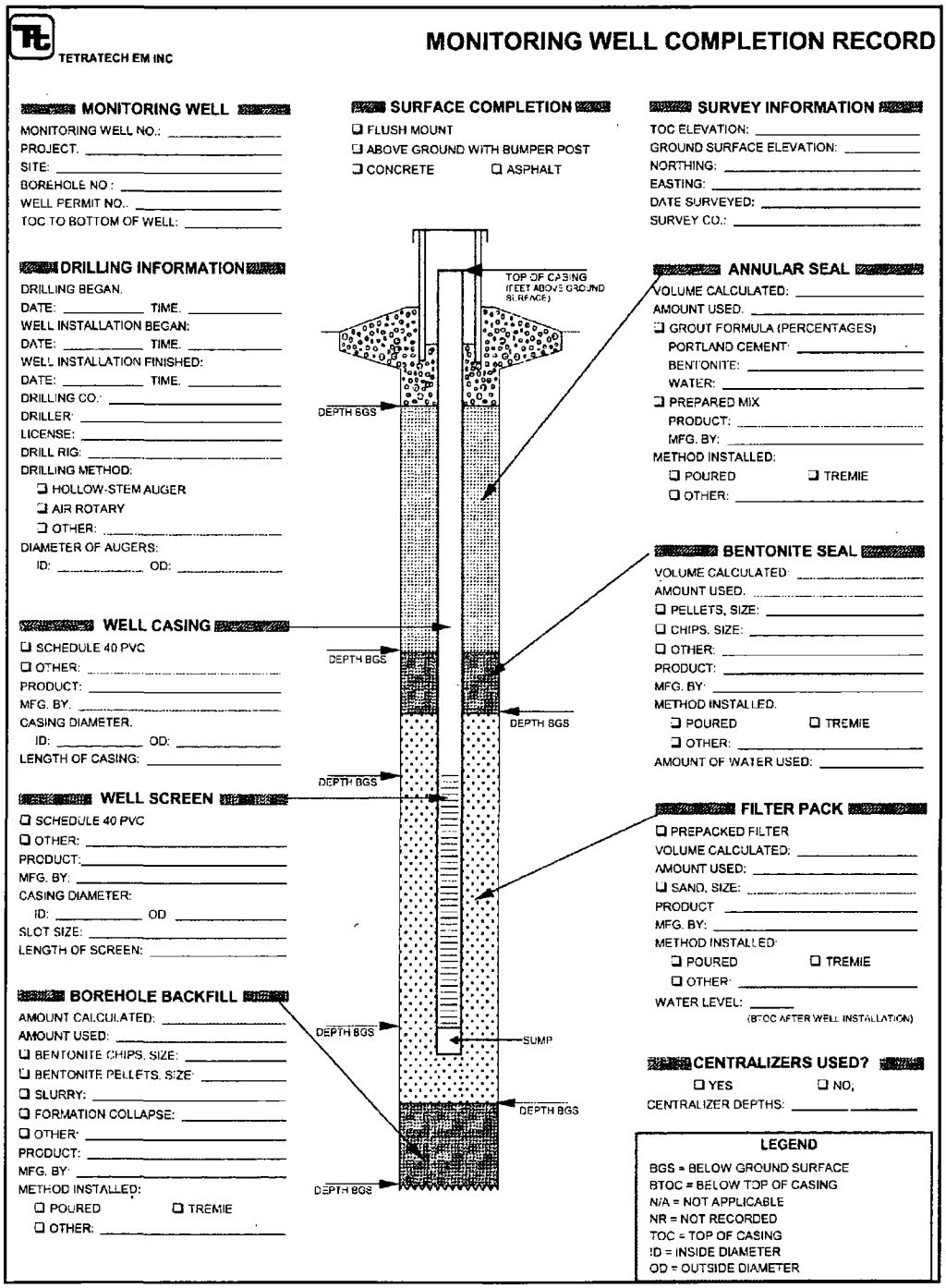


FIGURE 2

MONITORING WELL CONSTRUCTION DIAGRAM

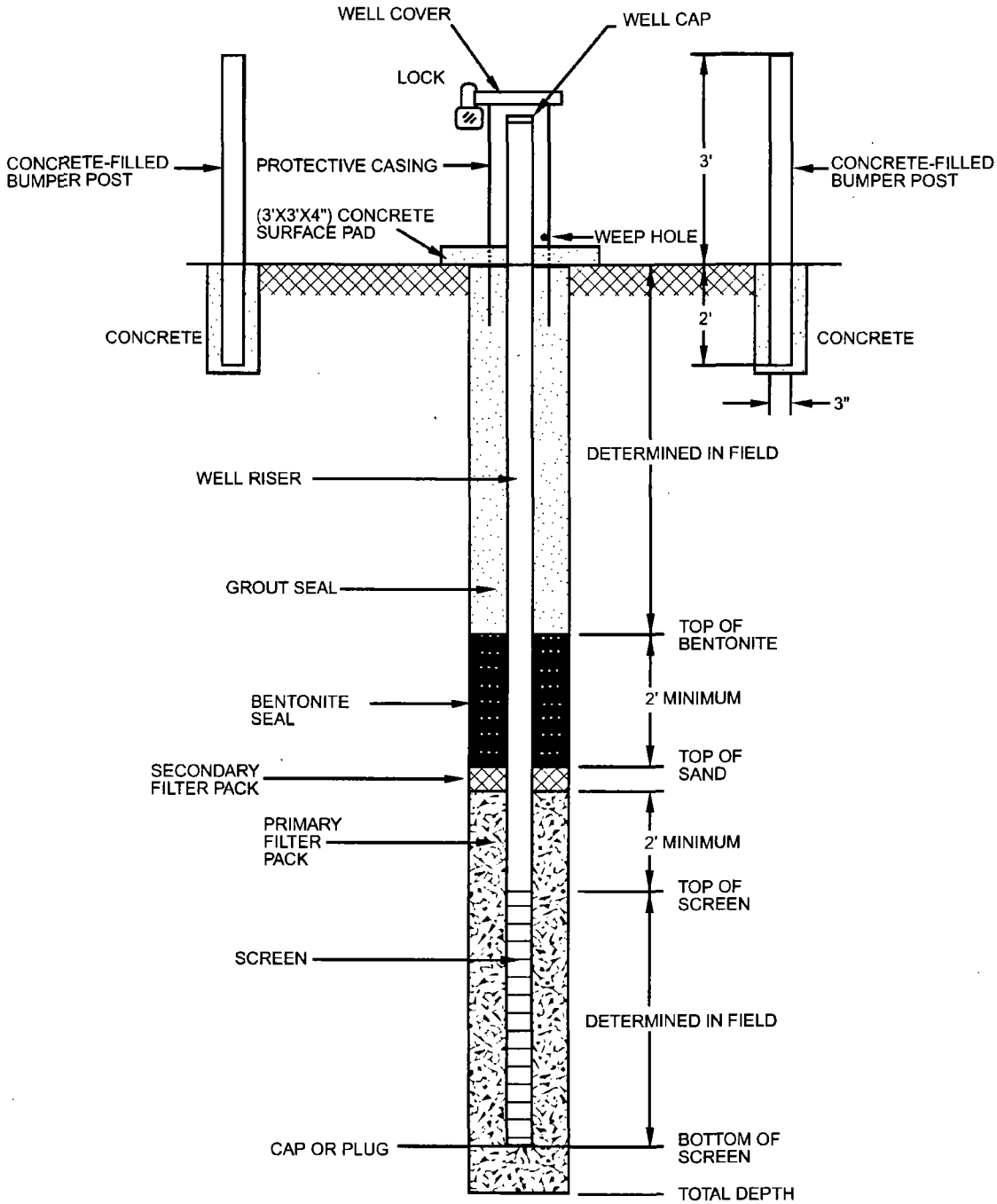
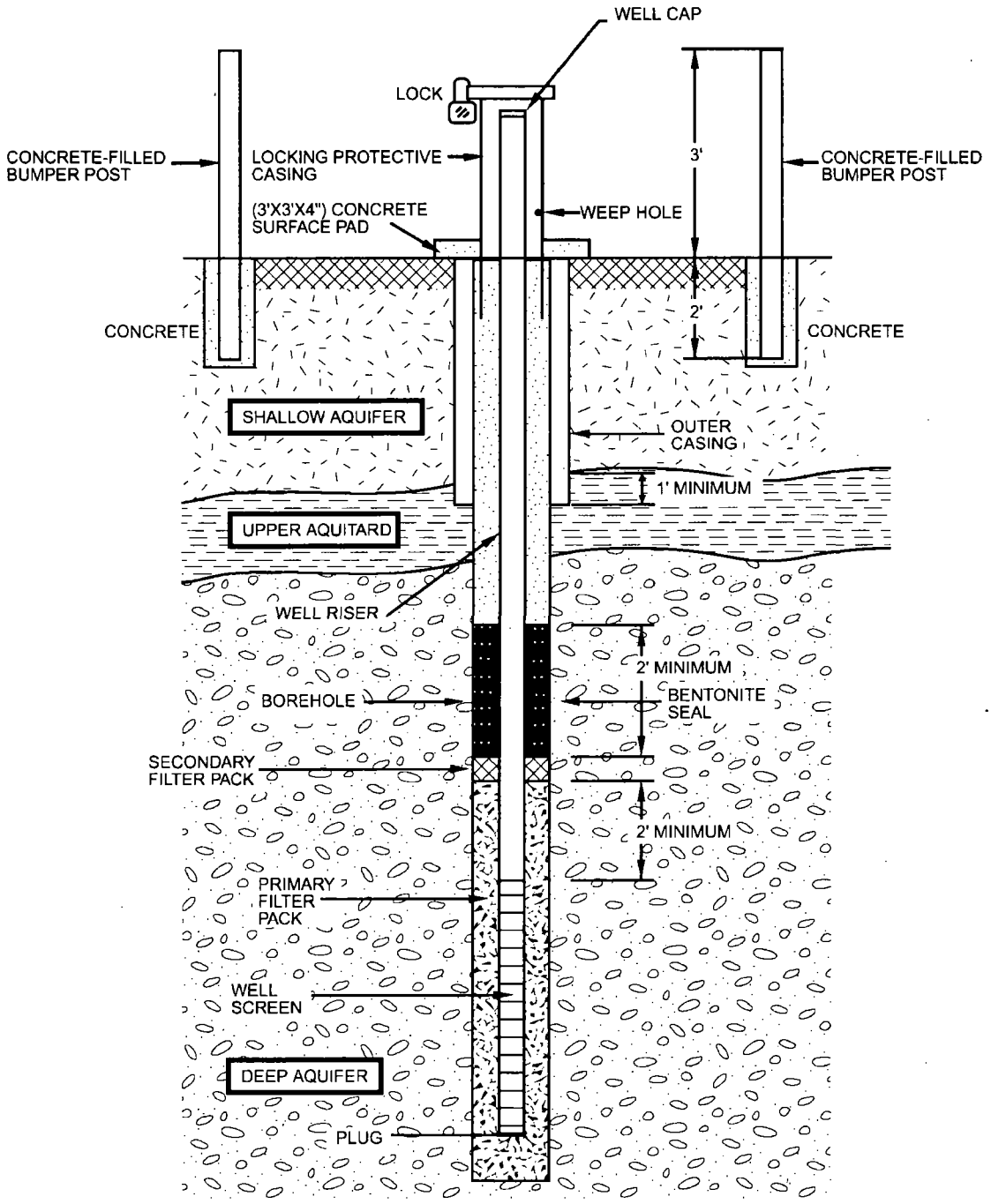


FIGURE 3

MULTIPLE CASING WELL CONSTRUCTION DIAGRAM



SOP APPROVAL FORM

PRC ENVIRONMENTAL MANAGEMENT, INC.

STANDARD OPERATING PROCEDURE

BOREHILL DRILLING - HOLLOW STEM AUGER DRILLING

SOP NO. 045

REVISION NO. 1

Approved by:

Quality Assurance Officer

Date

1.0 BACKGROUND

1.1 PURPOSE

This standard operating procedures (SOP) provides general guidance for the planning and implementation of hollow-stem auger geologic drilling for field investigations of hazardous waste sites. This SOP is not intended for use in site-specific situations. Consult site project plan for step by step procedures.

1.2 SCOPE

Some general considerations should be followed when using the hollow stem auger method. These involve borehole advancement, lithologic sampling, well installation, decontamination procedures, contaminated soil containment, borehole abandonment and health and safety monitoring.

1.3 REFERENCES

Acker, W. L. Basic Procedures for Soil Sampling and Core Drilling. Scranton, Penn: Acker Drill Co., 1976

Barcelona, M.J., J.P. Gibbs, J.A. Helfrid, and E.E. Garske, Practical Guide for Groundwater Sampling. SWS Contract Report 324, Champaign, Illinois: Illinois State Water Survey, 1985

Johnson Division, VOP WC., Groundwater and Wells, St. Paul, Minnesota 1980

National Water Well Association, Water Well Specifications, Berkely, California: Premier Press 1981

1.4 REQUIREMENTS AND RESOURCES

The following equipment are required for hollow stem auger drilling:

- Hollow Stem Auger Drill Rig (w/Associated Drill Tools and Hardware)
- Qualified and Experienced Driller and Helpers

2.0 PROCEDURES

The following procedures should be followed to operate the hollow stem auger.

2.1 GENERAL OPERATION

Hollow-stem augers are used to advance the borehole when discrete soil samples are needed. The augers are advanced by applying downward pressure on the augers as they are rotated. Material is forced to the ground surface around the exterior of the auger (spiral flights bring soil to the ground surface) during drilling. Cuttings are brought to the surface and can be identified. The hollow-stem auger consists of (1) a hollow steel tube with 5-foot spiral flights (internal diameters (IDs) range from 2-1/2 to 13-inches) and (2) a finger-type cutter head at the bottom of the lead auger (drill rods can be removed or inserted through the center of the auger assembly, facilitating soil sampling). A bottom plug can be inserted into the hollow center of the cutter head to prevent loose (unconsolidated) soil from coming up into the auger. This plug also has a pilot cutting head. The plug may be removed from the auger whenever a soil sample from below the cutter head is needed. Lithologic sampling is accomplished by removing the center plug from the auger stem and placing the appropriate sampling device (for example, split-spoon, or Shelby tube (see SOP 005)) at the end of the drill-rod assembly. The drive stem is

inserted through the hollow-stem auger and driven ahead of the augered material by a rig mounted, weighted hammer. The boreholes may be completed as wells or abandoned.

2.1.1. Monitoring Well Installation

Well installation is described in more detail in SOP 020. However, specific well completion methods should be followed when using the hollow-stem auger method. Borehole advancement will proceed to the desired depth. Upon reaching the desired depth, a small-diameter casing and screen can be set inside the hollow stem to produce a monitoring well. The augers are removed by section while the well screen and risers are held in place. Typically, one 5-foot section of auger is removed at a time. Clean sand and gravel pack is installed as the augers are withdrawn. Once the screen is properly covered (usually to 2 feet above the top of the screen), a clay (bentonite) seal should be installed (at least 2 feet of bentonite pellets or pressure grouted bentonite slurry should be placed on top of the filter pack.) As a final step, grout or other impermeable material is tremied in place on top of the clay seal to ground level as the remaining auger sections are removed. Careful installation of clay or grout seals is essential, especially in areas where multiple aquifers are encountered. Aquifer cross-contamination will be avoided, if at all possible.

2.1.2 Borehole Abandonment

In the event that a borehole or well needs to be abandoned, the hole will be filled from the bottom to the surface using a cement-bentonite grout. The cement-bentonite grout will be of the same consistency used to seal off the annular space of a completed well. The depth of the hole will be measured with a steel tape and the volume of grout calculated to plug the hole. The

method used to place the grout into the hole will ensure that it is filled from the bottom to the surface (e.g. tremie pipe). Cement-bentonite grout will be pumped into the hole until it rises to within 5 feet of the land surface. It will then be allowed to set overnight before the remainder of the hole is filled with neat cement. An alternative to cement-bentonite grout is straight bentonite grout. A cement cap will be made over the abandoned borehole that will allow any surface water to drain away from the area. The area of the abandoned borehole will be marked for future reference.

2.2 CONSIDERATIONS

Hollow-stem augering allows for drilling through soils below the water table with minimal cross-contamination. However, sediments such as flowing sands may "blow up" into the augers. If this situation is encountered (or will be known to occur), water of known chemical quality may be used to control sediment inflow. This method provides a greater head on the drilled sediments and prevents materials from advancing up through the auger.

The borehole should be advanced without using water unless absolutely necessary. Use of water while advancing through the unsaturated zone should not be necessary. If water is added during boring, it should be obtained from a source that has been analyzed and shows no contamination. It may be advisable to verify the quality of water through testing or by consulting local water authorities. If a sample is to be obtained, the borehole will be advanced by alternately boring a specified interval, removing the drive stem and bit, reinserting the drive stem with a sampler attached, and obtaining a sample. This procedure is continued until the desired depth is reached.

The auger stem, drive stem, and bit will be decontaminated prior to boring at a borehole location. At a minimum, this will include steam cleaning of the auger and drive stem. If a discrete zone of contamination is encountered during drilling, and drilling is to advance through this zone, downhole tools in contact with the contaminated zone will be steam cleaned after boring has proceeded through the contaminated interval. Additionally, to prevent downhole cross-contamination, it may be advisable to install an outer casing, grout the borehole while withdrawing the auger, redrill the hole through the grout using an auger with a smaller diameter auger, and then further advance the borehole.

Contaminated soil or water encountered during boring activities can be containerized within 55-gallon drums at the surface upon removal from the borehole.

Specific health and safety considerations will vary depending on site-specific factors. These considerations will generally be specifically detailed in a work plan, quality assurance project plan (QAPjP), or health and safety plan.

2.3 ADVANTAGES AND DISADVANTAGES

The hollow-stem auger method is the method of choice under the following circumstances:

- The boring is generally less than 100 feet in depth (see Table 1 for drilling method limitations). Depths exceeding 100 feet should be discussed with the drilling contractor.
- Boring is through a single saturated zone or through zones where cross-contamination is not suspected to be a potential problem.
- Locations of ground-water levels are important.

- Drilling must proceed with minimal use of water or boring fluid (little or no water is needed).
- Information concerning depth-discrete downhole contamination or water bearing zones can be easily obtained.
- Small-diameter monitoring wells are to be installed.
- Intact or undisturbed lithologic samples are required.
- Drilling will proceed through unconsolidated or loosely consolidated materials (the augers act like casing).
- Boring of monitoring wells needs to be completed in a short time period.

The disadvantages of the method are as follows:

- There is potential for cross-contamination from higher to lower hydrostratigrahic units.
- The method generally cannot be used for wells deeper than 100 feet (see Table 1 for drilling method limitations).
- The method is difficult to use when large cobbles or flowing sands are encountered.
- The method cannot be used to drill through competent bedrock.
- Hollow-stem augering may smear natural clays into open sands and gravels, thus limiting free flow of fluids into the wells and decreasing the response to hydraulic tests.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

MONITORING WELL DEVELOPMENT

SOP NO. 021

REVISION NO. 3

Last Reviewed: October 2000

Quality Assurance Approved

October 5, 2000

Date

1.0 BACKGROUND

All drilling methods impair the ability of an aquifer to transmit water to a drilled hole. This impairment is typically a result of disturbance of soil grains (smearing) or the invasion of drilling fluids or solids into the aquifer during the drilling process. The impact to the hydrologic unit surrounding the borehole must be remediated so that the well hydraulics and samples collected from the monitoring well are representative of the aquifer.

Well development should be conducted as an integral step of monitoring well installation to remove the finer-grained material, typically clay and silt, from the geologic formation near the well screen and filter pack. (Monitoring well installation is discussed in standard operating procedure [SOP] No. 020.) The fine-grained particles may interfere with water quality analyses and alter the hydraulic characteristics of the filter pack and the hydraulic unit adjacent to the well screen. Well development improves the hydraulic connection between water in the well and water in the formation. The most common well development methods are surging, jetting, overpumping, and bailing.

The health and safety plan for the site should be followed to avoid exposure to chemicals of concern. Water, sediment, and other waste removed from a monitoring well should be disposed of in accordance with applicable federal, state, and local requirements.

1.1 PURPOSE

This SOP establishes the requirements and procedure for monitoring well development. Well development improves the hydraulic characteristics of the filter pack and borehole wall by performing the following functions:

- Reducing the compaction and the intermixing of grain sizes produced during drilling by removing fine material from the pore spaces.
- Removing the filter cake or drilling fluid film that coats the borehole as well as much or all of the drilling fluid and natural formation solids that have invaded the formation.
- Creating a graded zone of sediment around the screen, thereby stabilizing the formation so that the well can yield sediment-free water.

1.2 SCOPE

This SOP applies to the development of newly installed monitoring wells. The SOP identifies the most commonly used well development methods; these methods can be used individually or in combination to achieve the most effective well development. Selection of a particular method will depend on site conditions, equipment limitations, and other factors. The method selected and the rationale for selection should be documented in a field logbook or appropriate project reports.

1.3 DEFINITIONS

Aquifer: A geologic formation, group of formations, or part of a formation that is saturated and capable of storing and transmitting water.

Aquitard: a geologic formation, group of formations, or part of a formation through which virtually no water moves.

Bailer: A cylindrical sampling device with valves on either end, used to extract water from a well or borehole.

Bentonite seal: A colloidal (extremely fine particle that will not settle out of solution) clay seal separating the sand pack from the surface seal.

Drilling fluid: A fluid (liquid or gas) that may be used in drilling operations to remove cuttings from the borehole, to clean and cool the drill bit, and to maintain the integrity of the borehole during drilling.

Filter pack: A clean, uniform sand or gravel placed between the borehole wall and the well screen to prevent formation material from entering the screen.

Grout seal: A fluid mixture of (1) cement and water or (2) cement, bentonite, and water that is placed above the bentonite seal between the casing and the borehole wall to secure the casing in place and keep water from entering the borehole.

Hydraulic conductivity: A measure of the ease with which water moves through a geologic formation. Hydraulic conductivity, K, is typically measured in units of distance per time in the direction of groundwater flow.

Hydrologic units: Geologic strata that can be distinguished on the basis of capacity to yield and transmit fluids. Aquifers and confining units are types of hydrologic units.

Oil air filter: A filter or series of filters placed in the airflow line from an air compressor to reduce the oil content of the air.

Oil trap: A device used to remove oil from the compressed air discharged from an air compressor.

Riser: The pipe extending from the well screen to or above the ground surface.

Specific conductance: A measure of the ability of the water to conduct an electric current. Specific conductance is related to the total concentration of ionizable solids in the water and is inversely proportional to electrical resistance.

Static water level: The elevation of the top of a column of water in a monitoring well or piezometer that is not influenced by pumping or conditions related to well installation, hydrologic testing, or nearby pumpage.

Transmissivity: The volume of water transmitted per unit width of an aquifer over the entire thickness of the aquifer flow, under a unit hydraulic gradient.

Well screen: A cylindrical pipe with openings of a uniform width, orientation, and spacing used to keep materials other than water from entering the well and to stabilize the surrounding formation.

Well screen jetting (hydraulic jetting): A jetting method used for development; nozzles and a high pressure pump are used to force water outwardly through the screen, the filter pack, and sometimes into the adjacent geologic unit.

1.4 REFERENCES

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1.5 REQUIREMENTS AND RESOURCES

The type of equipment used for well development will depend on the well development method. Well development methods and the equipment required are discussed in Section 2.1 of this SOP. In general, monitoring wells should be developed shortly after they are installed but no sooner than 24 hours after the placement of the grout seal, depending on the grout cure rate and well development method. Most drilling or well development rigs have pumps, air compressors, bailers, surge blocks, and other equipment that can be used to develop a monitoring well.

All downhole equipment should be properly decontaminated before and after each well is developed. See SOP No. 002 (General Equipment Decontamination) for details.

2.0 WELL DEVELOPMENT PROCEDURES

This section describes common well development methods, factors to be considered in selecting a well development method, procedures for initiating well development, well development duration, and calculations typically made during well development. In addition to this, procedures described in any work plans for well development should be fully consistent with local and state regulations and guidelines.

2.1 WELL DEVELOPMENT METHODS

Well development methods vary with the physical characterization of hydrologic units in which the monitoring well is screened and the drilling method used. The most common methods include mechanical surging, overpumping, air lift pumping, backwashing, surge bailing, and well jetting. These methods may be effective alone or may need to be combined (for example, overpumping combined with backwashing). Factors such as well design and hydrogeologic conditions will determine which well development method will be most practical and cost-effective. Commonly used well development methods are described in Sections 2.1.1 through 2.1.6.

The use of chemicals for monitoring well development should be avoided as much as possible. Introduction of chemicals may significantly alter groundwater chemistry in and around the well.

2.1.1 Mechanical Surging

The mechanical surging method forces water to flow in and out of the well screen by operating a plunger (or surge block) in the casing, similar to a piston in a cylinder. A typical surge block is shown in Figure 1. The surge block should fit snugly in the well casing to increase the surging action. The surge block is attached to a drill rod or drill stem and is of sufficient weight to cause the block to drop rapidly on the down stroke, forcing water contained in the borehole into the aquifer surrounding the well. In the recovery stroke or upstroke, water is lifted by the surge block, allowing water and fine sediments to flow back into the well from the aquifer. Down strokes and recovery strokes are usually 3 to 5 feet in length.

The surge block should be lowered into the water column above the well screen. The water column will effectively transmit the action of the block to the filter pack and hydrologic unit adjacent to the well screen. Development should begin above the screen and move progressively downward to prevent the surge block from becoming sand locked in the well. The initial surging action should be relatively gentle, allowing any material blocking the screen to break up, go into suspension, and then move into the well. As water begins to move easily both in and out of the screen, the surge block is usually lowered in increments to a level just above the screen. As the block is lowered, the force of the surging movement should be increased. In wells

equipped with long screens, it may be more effective to operate the surge block in the screen to concentrate its actions at various levels.

A pump or bailer should be used periodically to remove dislodged sediment that may have accumulated at the bottom of the well during the surging process. The pump or bailer should be moved up and down at the bottom of the well to suspend and collect as much sediment as possible.

The accumulation of material developed from a specific screen interval can be measured by sounding the total depth of the well before and after surging. Continue surging until little or no sand accumulates.

2.1.2 Overpumping

Overpumping involves pumping the well at a rate substantially higher than it will be pumped during well purging and groundwater sampling. This method is most effective on coarse-grained formations and is usually conducted in conjunction with mechanical surging or backwashing. Overpumping is commonly implemented using a submersible pump. In cases were the water table is less than 30 feet from the top of the casing, it is possible to overpump the well with a centrifugal pump. The intake pipe is lowered into the water column at a depth sufficient to ensure that the water in the well is not drawn down to the pump intake level. The inflow of water at the well screen is not dependent on the location of the pump intake as long as it remains submerged.

Overpumping will induce a high velocity water flow, resulting in the flow of sand, silt, and clay into the well, opening clogged screen slots and cleaning formation voids and fractures. The movement of these particles at high flow rates should eliminate particle movement at the lower flow rates used during well purging and sampling. The bridging of particles against the screen because of the flow rate and direction created by overpumping may be overcome by using mechanical surging or backwashing in conjunction with this method.

2.1.3 Air Lift Pumping

Air lift pumping uses a two-pipe system consisting of an air injection pipe and a discharge pipe. In this well development method, an air lift pump is operated by cycling the air pressure on and off for short periods of time. This operation provides a surging action that can dislodge fine-grained particles in the vicinity of the well screen. Subsequently applying a steady low pressure removes the fines drawn into the well by the surging action.

The bottom of the air lift should be at least 10 feet above the top of the well screen. Air is injected through an inner pipe at sufficient pressure to bubble out directly into the surrounding discharge pipe. The bubbles formed by the injected air cause the column of water in the discharge pipe to be lifted upward and allow water from the aquifer to flow into the well. This arrangement prevents injected air from entering the well screen. Pumping air through the well screen and into the filter pack and adjacent hydrologic unit should be avoided because it can cause air entrainment, inhibiting future sampling efforts and possibly altering groundwater chemistry.

The air injected into the well should be filtered using an oil/air filter and oil trap to remove any compressor lubricant entrained in the air. Air pressures required for this well development method are relatively low; an air pressure of 14.8 pounds per square inch should move a 30-foot column of water. For small-diameter, shallow wells where the amount of development water is likely to be limited, tanks of inert gas (such as nitrogen) can be used as an alternative to compressed air.

2.1.4 Backwashing

Effective development procedures should cause flow reversals through the screen openings that will agitate the sediment, remove the finer fraction, and then rearrange the remaining formation particles. Backwashing overcomes the bridging that results from overpumping by allowing the water that is pumped to the top of the well to flow back through the submersible pump and out through the well screen. The backflow portion of the backwashing cycle breaks down bridging, and the inflow then moves the fine material toward the screen and into the well.

Some wells respond satisfactorily to backwashing techniques, but the surging effect is not vigorous enough to obtain maximum results in many cases.

A variation of backwashing may be effective in low-permeability formations. After the filter pack is installed on a monitoring well, clean water is circulated down the well casing, out through the well screen and filter pack, and up through the open borehole before the grout or bentonite seal is placed in the annulus. Flow rates should be controlled to prevent floating the filter pack. Because of the low hydraulic conductivity of the formation, negligible amounts of water will infiltrate into the formation. Immediately after this procedure, the bentonite seal should be installed, and the nonformation water should be pumped out of the well and filter pack.

2.1.5 Surge Bailing

Surge bailing can be an effective well development method in relatively clean, permeable formations where water flows freely into the borehole. A bailer made of stainless steel or polyvinyl chloride and slightly smaller than the well casing diameter is allowed to fall freely through the borehole until it strikes the groundwater surface. The contact of the bailer produces a downward force and causes water to flow outward through the well screen, breaking up bridging that has developed around the screen. As the bailer fills and is rapidly withdrawn from the well, the drawdown created causes fine particles to flow through the well screen and into the well. Subsequent bailing can remove these particles from the well. Lowering the bailer to the bottom of the well and using rapid short strokes to agitate and suspend solids that have settled to the well bottom can enhance removal of sand and fine particles. Bailing should continue until the water is free of suspended particles.

2.1.6 Well Jetting

Well jetting can be used to develop monitoring wells in both unconsolidated and consolidated formations. Water jetting can open fractures and remove drilling mud that has penetrated the aquifer. The discharge force of the jetting tool is concentrated over a small area of the well screen. As a result, the tool must be rotated constantly while it is raised and lowered in a very small increments to be sure that all portions of the screen are exposed to the jetting action.

Jetting is relatively ineffective on the fine screens typically used in monitoring wells (slot sizes from 0.01 to 0.02 inch). In addition, jetting requires the introduction of external water into the well and surrounding formation. This water should be obtained from a source of known chemistry. Water introduced for development should be completely removed from the aquifer immediately after development.

The use of compressed air as a jetting agent should not be employed for development of monitoring wells. Compressed air could entrain air in the formation, introduce oil into the formation, and damage the well screen.

2.2 FACTORS TO CONSIDER WHEN SELECTING A WELL DEVELOPMENT METHOD

It is important to check federal, state, and local regulatory requirements for monitoring well development requirements. This SOP may be changed to accommodate applicable regulations, site conditions, or equipment limitations.

The type of geologic material, the design and completion of the well, and the type of drilling method used are all factors to be considered during the development of a monitoring well.

Monitoring well development should usually be started slowly and gently and then performed with increasing vigor as the well is developed. Most well development methods require the application of sufficient energy to disturb the filter pack, thereby freeing fine particles and allowing them to be drawn into the well. The coarser particles then settle around and stabilize the screen.

Development procedures for wells completed in fine sand and silt strata should involve methods that are relatively gentle so that strata material will not be incorporated into the filter pack. Vigorous surging for development can produce mixing of the fine strata and filter pack and produce turbid samples from the formation. In addition, development methods should be carefully selected based upon the potential contaminants present, the quantity of wastewater generated, and requirements for containerization or treatment of wastewater.

For small diameter and small volume wells, a development bailer can be used in place of a submersible pump in the pumping method. Similarly, a bailer can be used in much the same fashion as a surge block in small diameter wells.

Any time an air compressor is used for well development, it should be equipped with an oil air filter or oil trap to minimize the introduction of oil into the screened area. The presence of oil could impact the organic constituent concentrations of the water samples collected from the well.

The presence of light nonaqueous phase liquid (LNAPL) can impact monitoring well development. Water jetting or vacuum-enhanced well development may assist in breaking down the smear zone in the LNAPL. Normal development procedures are conducted in the water-saturated zone and do not affect the LNAPL zone.

2.3 INITIATING WELL DEVELOPMENT

Newly completed monitoring wells should be developed as soon as practical, but no sooner than 24 hours after grouting is completed if rigorous well development methods are used. Development may be initiated shortly after well installation if the development method does not interfere with the grout seal. State and local regulations should be checked for guidance. The following general well development steps can be used with any of the methods described in Section 2.1.

1. Assemble the necessary equipment on a plastic sheet around the well. This may include a water level meter (or oil/water interface probe if LNAPL or dense nonaqueous phase liquid is present); personal protective equipment; pH, conductivity, temperature, and turbidity meters; air monitoring equipment; Well Development Data Sheets (see Figure 2); a watch; and a field logbook.

2. Open the well and take air monitoring readings at the top of the well casing and in the breathing zone. See SOP No. 003 (Organic Vapor Air Monitoring) for additional guidance.

3. Measure the depth to water and the total depth of the monitoring well. See SOP No. 014 (Static Water Level, Total Well Depth, and Immiscible Layer Measurement) for additional guidance.

4. Measure the initial pH, temperature, turbidity, and specific conductance of the groundwater from the first groundwater that comes out of the well. Note the time, initial color, clarity, and odor of the water. Record the results on a Well Development Data Sheet (see Figure 2) or in a field logbook. See SOPs No. 011 (Field Measurement of Water Temperature), 012 (Field Measurement of pH), 013 (Field Measurement of Specific Conductance), and 088 (Field Measurement of Water Turbidity) for additional guidance.

5. Develop the well using one or more of the methods described in Section 2.1 until the well is free of sediments and the groundwater turbidity has reached acceptable levels. Record the development method and other pertinent information on a Well Development Data Sheet see Figure 2) or in a field logbook.

6. Containerize any groundwater produced during well development if groundwater contamination is suspected. The containerized water should be sampled and analyzed to determine an appropriate disposal method.

7. Do not add water to assist in well development unless the water is from a source of known chemical quality and the addition has been approved by the project manager. If water is added, five times the amount of water introduced should be removed during development.

8. Continue to develop the well, repeating the water quality measurements for each borehole volume. Development should continue until each water quality parameter is stable to within 10 percent. Development should also continue until all the water added during development (if any) is removed or the water has a turbidity of less than 50 nephelometric turbidity units. This level may only be attainable after allowing the well to settle and testing at low flow sampling rates.

9. At the completion of well development, measure the final pH, temperature, turbidity, and specific conductance of the groundwater. Note the color, clarity, and odor of the water. Record the results on a Well Development Data Sheet (see Figure 2) or in a field logbook. In addition to the final water quality parameters, the following data should be noted on the Well Development Data Sheet: well identification, date(s) of well installation, date(s) and time of well development, static water level before and after development, quantity of water removed and time of removal, type and capacity of pump or bailer used, and well development technique.

All contaminated water produced during development should be containerized in drums or storage vessels properly labeled with the date collected, generating address, well identification, and consultant contact number.

2.4 DURATION OF WELL DEVELOPMENT

Well development should continue until representative water, free of the drilling fluids, cuttings, or other materials introduced during well construction is obtained. When pH, temperature, turbidity, and specific conductance readings stabilize and the water is visually clear of suspended solids, the water is representative of formation water. The minimum duration of well development should vary in accordance with the method used to develop the well. For example, surging and pumping the well may provide a stable, sediment free sample in a matter of minutes, whereas bailing the well may require several hours of continuous effort to obtain a clear sample.

An on-site project geologist should make the final decision as to whether well development is complete. This decision should be documented on a Well Development Data Sheet (see Figure 2) or in a field logbook.

2.5 CALCULATIONS

It is necessary to calculate the volume of water in the well. Monitoring well diameters are typically 2, 3, 4, or 6 inches. The height of water column (in feet) in the well can be multiplied by the following conversion factors to calculate the volume of water in the well casing.

Well Diameter (inches)	Volume (gallon per foot)
2	0.1631
3	0.3670
4	0.6524
6	1.4680

3.0 POTENTIAL PROBLEMS

The following potential problems can occur during development of monitoring wells:

- In some wells the pH, temperature, and specific conductance may stabilize but the water remains turbid. When this occurs, the well may still contain construction materials (such as drilling mud in the form of a mud cake) and formation soils that have not been washed out of the borehole. Excessive or thick drilling muds cannot be flushed out of a borehole with one or two well volumes of flushing. Continuous flushing over a period of several days may be necessary to complete well development. If the well is completed in a silty zone, it may be necessary to sample with low flow methods or filtering.

- Mechanical surging and well jetting disturb the formation and filter pack more than other well development methods. In formations with high clay and silt contents, surging and jetting can cause the well screen to become clogged with fines. If an excessive amount of fines is produced, sand locking of the surge block may result. Well development with these methods should be initiated gently to minimize disturbance of the filter pack and to prevent damage to the well screen.

- Effective overpumping may involve the discharge of large amounts of groundwater. This method is not recommended when groundwater extracted during well development is contaminated with hazardous constituents. If the hazardous constituents are organic compounds, this problem can be partially overcome by passing the groundwater through an activated carbon filter.

- When a well is developed by mechanical surging or bailing, rapid withdrawal of the surge block or bailer can result in a large external pressure outside of the well. If the withdrawal is too rapid and this pressure is too great, the well casing or screen can collapse.

- A major disadvantage of well jetting is that an external supply of water is needed. The water added during well jetting may alter the hydrochemistry of the aquifer; therefore, the water added in this development procedure should be obtained from a source of known chemistry. In addition, the amount of water added during well development and the amount lost to the formation should be recorded.

- The use of air in well development can chemically alter the groundwater, either directly through chemical reaction or indirectly as a result of impurities introduced through the air stream. In addition, air entrainment within the formation can interfere with the flow of groundwater into the monitoring well. Consequently, air should not be injected in the immediate vicinity of the well screen.

FIGURE 1

SCHEMATIC DRAWING OF A SURGE BLOCK

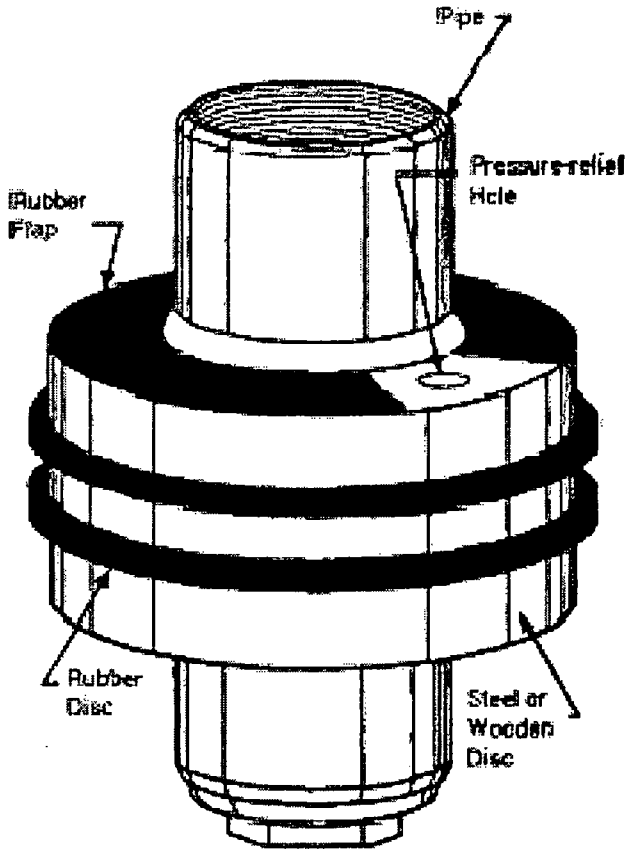

FIGURE 2

WELL DEVELOPMENT DATA SHEET

Sheet ___ of ___

WELL DEVELOPMENT DATA SHEET

BORING NO. _____ **WELL NO.** _____

Project _____
Project No. _____
Date(s) of Installation _____
Date(s) of Development _____
Personnel/Company _____
_____
Type of Rig Used _____

Casing Diameter/Type _____
Borehole Diameter _____
Screened Interval(s) _____
Total Length of Well Casing _____
Measured Total Depth (TOC) Initial _____
Final _____
Initial Depth to Water
(TOC) _____ Date _____ Time _____
Stabilized Depth to Water
(TOC) _____ Date _____ Time _____

DEVELOPMENT TECHNIQUE(S) EQUIPMENT TYPE/CAPACITY

___ Surging _____
___ Overpumping _____
___ Air Lift Pumping _____
___ Backwashing _____
___ Bailing _____
___ Well Jetting _____

FLUIDS ADDED

Lost Drilling Fluid: _____ Gallons
Lost Purge Water: _____ Gallons
Water During Installation: _____ Gallons
Total Fluids Added: _____ Gallons
Source of Added Water: _____
Sample Collected of Added Water: Y N
Sample Designation of Added Water: _____

PURGE VOLUME CALCULATION

Casing Volume: _____ Ft. of water
x _____ Gallons/Foot
= _____ Gallons per Single Casing Volume
Sand Pack Volume: _____ Ft. of Saturated Sand Pack
x _____ Gallons/Foot (borehole diameter)
= _____ Gallons (in borehole)
- _____ Gallons of Casing Volume
= _____ x 0.3 (Assuming porosity = 30%)
= _____ Gallons Within Sand Pack
Single Purge Volume: _____ Gallons (Casing Vol. + Sand Pack Vol. + Fluids Added)
Minimum Purge Volume: _____ Gallons
Actual Purge Volume: _____ Gallons
Volume Measured by: _____
Rate of Development _____ Gallons/Minute (Hour, Day)
Pumping Rate/Depth _____ @ _____ Ft. (Below Grd.)
Immiscible Phases Present: Y N Thickness _____

Development Criteria: _____

Total Volume Discharged	Rate of Discharge	Time	Temp	pH	Specific* Conductance	Turbidity (NTU)	D.O., Clarity, Odor, PID Readings, Other:

Development Completed at _____ Gallons Discharged. Date: _____ Time: _____

Personnel: _____
_____

* Specific Conductance readings temperature compensated to 25°C, if not, report temperatures at which reading obtained.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

STATIC WATER LEVEL, TOTAL WELL DEPTH, AND IMMISCIBLE LAYER MEASUREMENT

SOP NO. 014

REVISION NO. 0

Last Reviewed: December 1999

Quality Assurance Approved

July 20, 1994

Date

1.0 BACKGROUND

Measurement of static water level, total well depth, and any immiscible layers is necessary before a well can be sampled and groundwater flow direction can be determined. If an immiscible layer is present, its depth and thickness must be determined. In addition, the static water level and total depth of a monitoring well are needed to determine a purging volume.

1.1 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide guidelines for field personnel measuring static water levels and total water depths of monitoring wells or piezometers. This SOP also provides guidelines for measuring immiscible layers in such wells.

1.2 SCOPE

This SOP describes the methodologies for measuring static water level, total well depth, and immiscible layer depth and thickness.

1.3 DEFINITIONS

To clarify the methodologies presented in this SOP, the following definitions are presented:

Electrical Water Level Indicator: An electrical probe used to determine the depth to fluid. The probe has a light or sound alarm connected to an open circuit. The circuit is closed and the alarm is activated when the probe contacts a conducting fluid such as water.

Immiscible Layer: A liquid phase that cannot be uniformly mixed or blended with water. Heavy immiscible phases sink in water; light immiscible phases float on water.

Interface Probe: An electrical probe used to determine the thicknesses of light or dense immiscible layers in the water column of a monitoring well.

Ionization Detector: A photoionization detector (PID) or a flame ionization detector (FID) is used to measure the level of volatile organic compounds in the gaseous phase. These units are generally not compound-specific and thus measure only total volatile organic compounds. The PID generally cannot detect as complete a range of compounds as the FID. This difference is the result of the relative ionization energies of the two detectors. Most PIDs cannot detect methane, but FIDs can. The HNu and Microtip are examples of PIDs; the Foxboro organic vapor analyzer (OVA) is an example of an FID.

Static Water Level: The level of water in a monitoring well or piezometer. This level can be measured as the depth to water or as the elevation of water relative to a reference mark or datum.

Total Well Depth: The distance from the ground surface to the bottom of a monitoring well or piezometer

1.4 REFERENCES

SOP No. 002, General Equipment Decontamination

U.S. Environmental Protection Agency. 1994. "Water Level Measurement." Environmental Response Team SOP #2043 (Rev. #0.0, 10/03/94). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

The equipment required for measuring static water levels, total well depths, and immiscible layers is as follows:

- Electrical water level indicator
- Interface probe
- PID or FID

2.0 PROCEDURES

This section provides general guidance followed by specific procedures for static water level, total well depth, and immiscible layer measurement.

Techniques for measuring depth to water and depth to the bottom of a monitoring well should be identified in the planning stage of field work. Also at this stage, measuring devices should be chosen, and an individual should be assigned to take and record measurements.

All measurement instruments should be decontaminated before and after use and between measurement locations. Refer to SOP No. 002, General Equipment Decontamination.

Before initiating any measuring activities, the ambient air at a monitoring well head should be monitored for possible emissions of volatile organic compounds. To accomplish this monitoring, a PID or an FID should be used. The health and safety plan for on-site activities should provide action levels and the rationale for selection of either detector.

Appropriate respiratory protection equipment should be worn by the sampling team. Wells should be approached from the upwind side. When opening the monitoring well, the sampling team should systematically survey the inside of the well casing, the area from the casing to the ground, the area from above the well casing to the breathing zone, and the area around the well. Readings for comparison to action levels should be taken not within the well casing but in the breathing zone. If PID or FID readings of volatile organic compounds are above action levels, the sampling team should retreat to a safe area and put on appropriate safety gear. The site-specific health and safety plan should be consulted for action levels.

2.1 STATIC WATER LEVEL MEASUREMENT

The procedure described below should be followed to measure the static water level in a monitoring well or piezometer.

An electric water level indicator is typically used for static water level measurement. The electrical probe of the indicator should be lowered into the monitoring well until the light or sound alarm is activated, indicating that the probe has touched the water surface. The static water level should then be read directly from the indicator to the 0.01-foot fraction. If the monitoring well top is not flush with the ground surface, the distance between the static water level and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth to static water below ground surface. If surveyed elevations are available, they should be used to establish the water level elevation. To ensure measurement accuracy, the probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the values should be averaged. The measurement date and time, individual readings, and the average of the readings should be recorded in a field logbook.

2.2 TOTAL WELL DEPTH MEASUREMENT

The procedure described below should be followed to measure total well depth in a monitoring well or piezometer.

Total well depth measurement can be performed also using an electric water level indicator. The electrical probe of the indicator should be lowered into the monitoring well until resistance is met, indicating that the probe has reached the bottom of the well. The total well depth should then be read directly from the indicator to the 0.01-foot fraction. If the monitoring well top is not flush with the ground surface, the distance between the bottom of the well and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth from ground surface to the bottom of the well. To ensure measurement accuracy, the probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the values should be averaged. The measurement date and time, individual readings, and the average of the readings should be recorded in a field logbook.

2.3 IMMISCIBLE LAYER DETECTION AND MEASUREMENT

The procedure described below should be followed to detect and measure an immiscible layer in a monitoring well.

A light immiscible layer in a monitoring well can be detected by slowly lowering an interface probe to the surface of the water in the well. When the audible alarm sounds, the depth of the probe should be recorded. If the alarm is continuous, a light immiscible layer has been detected. To measure the thickness of this layer, the probe should then be lowered until the alarm changes to an oscillating signal. The oscillating alarm indicates that the probe has reached a water layer. The probe depth at the time the alarm begins oscillating should be recorded as the depth to water. The thickness of the light immiscible layer should then be determined by subtracting the depth at which a continuous alarm occurred from the depth at which the alarm began to oscillate. To ensure measurement accuracy, the interface probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the depths and thicknesses measured should be averaged. The measurement date and time, individual readings for depth and thickness, and average values for depth and thickness should be recorded in a field logbook.

To determine whether a dense immiscible layer is present, the interface probe should be lowered further into the monitoring well. If the alarm changes from an oscillating to a continuous signal, a heavier immiscible layer has been detected, and the probe depth should be recorded at that point. Total well depth obtained in Section 2.2 should be used for calculating the thickness of the dense layer. The dense layer should be calculated by subtracting the depth at which the alarm became continuous from the total well depth. This procedure provides an estimate of the thickness of the dense layer in the monitoring well. To ensure measurement accuracy, the interface probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the depths and thicknesses measured should be averaged. The measurement date and time, individual readings for depth and thickness, and average values for depth and thickness should be recorded in a field logbook.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GROUNDWATER SAMPLE COLLECTION USING MICROPURGE TECHNOLOGY

SOP NO. 015

REVISION NO. 0

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Date

1.0 BACKGROUND

Groundwater sample collection is an integral part of site characterization at many contaminant release investigation sites. Often, a requirement of groundwater contaminant investigation is to evaluate contaminant concentrations in the aquifer. Since data quality objectives of most investigations require a laboratory setting for chemical analysis, samples must be collected from the aquifer and submitted to a laboratory for analysis. Therefore, sample collection and handling must be conducted in a manner that minimizes alteration of chemical characteristics of the groundwater.

In the past, most sample collection techniques followed federal and state guidance. Acceptable protocol included removal of water in the casing of a monitoring well (purging), followed by sample collection. The water in the casing was removed so groundwater from the formation could flow into the casing and be available for sample collection. Sample collection was commonly completed with a bailer, bladder pump, controlled flow impeller pump, or peristaltic pump. Samples were preserved during collection. Often, samples to be analyzed for metals contamination were filtered through a 0.45-micron filter prior to preservation and placement into the sample container.

Research conducted by several investigators has demonstrated that a significant component of contaminant transport occurs while the contaminant is sorbed onto colloid particles. Colloid mobility in an aquifer is a complex, aquifer-specific transport issue, and its description is beyond the scope of this Standard Operating Procedure (SOP). However, concentrations of suspended colloids have been measured during steady state conditions and during purging activities. Investigation results indicate standard purging procedures can cause a significant increase in colloid concentrations, which in turn may bias analytical results.

Micropurge sample collection provides a method of minimizing increased colloid mobilization by removing water from the well at the screened interval at a rate that preserves or minimally disrupts steady-state flow conditions in the aquifer. During micropurge sampling, groundwater is discharged from the aquifer at a rate that the aquifer will yield without creating a cone of depression around the sampled well. Research indicates that colloid mobilization will not increase above steady-state conditions during low-flow discharge. Therefore, the collected sample is more likely to represent steady-state groundwater chemistry.

1.1 PURPOSE

The purpose of this SOP is to describe the procedures to be used to collect a groundwater sample from a well using the micropurge technology. The following sections describe the equipment to be used and the methods to be followed to promote uniform sample collection techniques by field personnel that are experienced in sample collection and handling for environmental investigations.

1.2 SCOPE

This SOP applies to groundwater sampling using the micropurge technology. It is intended to be used as an alternate SOP to the general "Groundwater Sampling" SOP (SOP No. 10) that provides guidance for the general aspects of groundwater sampling.

1.3 DEFINITIONS

Colloid: Suspended particles that range in diameter from 5 nanometers to 0.2 micrometers.

Dissolved oxygen: The ratio of the concentration or mass of oxygen in water relative to the partial pressure of gaseous oxygen above the liquid which is a function of temperature, pressure, and concentration of other solutes.

Flow-through cell: A device connected to the discharge line of a groundwater purge pump that allows regular or continuous measurement of selected parameters of the water and minimizes contact between the water and air.

pH: The negative base-10 log of the hydrogen-ion activity in moles per liter.

Reduction and oxidation potential: A numerical index of the intensity of oxidizing or reducing conditions within a system, with the hydrogen-electrode potential serving as a reference point of zero volts.

Specific conductance: The reciprocal of the resistance in ohms measured between opposite faces of a centimeter cube of aqueous solution at a specified temperature.

Turbidity: A measurement of the suspended particles in a liquid that have the ability to reflect or refract part of the visible portion of the light spectrum.

1.4 REFERENCES

Puls, R. W. and M. J. Barcelona. 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. U.S. Environmental Protection Agency. Office of Research and Development. EPA/540/S-95/504. April.

1.5 REQUIREMENTS AND RESOURCES

The following equipment is required to complete micropurge sample collection :

- Water level indicator
- Adjustable flow rate pump (bladder, piston, peristaltic, or impeller)
- Discharge flow controller
- Flow-through cell
- pH probe
- Dissolved oxygen (DO) probe
- Turbidity meter
- Oxidation and reduction (Redox or Eh) probe
- Specific conductance (SC) probe (optional)
- Temperature probe (optional)
- Meter to display data for the probes
- Calibration solutions for pH, SC, turbidity, and DO probes, as necessary
- Container of known volume for flow measurement or calibrated flow meter
- Data recording and management system

2.0 PROCEDURE

The following procedures and criteria were modified from the U. S. Environmental Protection Agency guidance titled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (Puls and Barcelona 1996). This reference may be consulted for a more detailed description of micropurge sampling theory.

Micropurging is most commonly accomplished with low-discharge rate pumps, such as bladder pumps, piston pumps, controlled velocity impeller pumps, or peristaltic pumps. Bailers and high capacity submersible pumps are not considered acceptable micropurge sample collection devices. The purged water is monitored (in a flow-through cell or other constituent monitoring device) for chemical and optical parameters that indicate steady state flow conditions between the sample extraction point and the aquifer. Samples are collected when steady state conditions are indicated.

Groundwater discharge equipment may be permanently installed in the monitoring well as a dedicated system, or it can be installed in each well as needed. Most investigators agree that dedicated systems will provide the best opportunity for collecting samples most representative of steady state aquifer conditions, but the scope of a particular investigation and available investigation funds will dictate equipment selection.

2.1 EQUIPMENT CALIBRATION

Prior to sample collection, the monitoring equipment used to measure pH, Eh, DO, turbidity, and SC should be calibrated or checked according to manufacturer's directions. Typically, calibration activities are completed at the field office at the beginning of sampling activities each day. The pH meter calibration should bracket the pH range of the wells to be sampled (acidic to neutral pH range [4.00 to 7.00] or neutral to basic pH range [7.00 to 10.00]). The DO meter should be calibrated to one point (air-saturated water) or two points (air-saturated water and water devoid of all oxygen). The SC meter cannot be calibrated in the field. It is checked against a known standard (typical standards are 1, 10, and 50 millimhos per centimeter at 25 • C). The offset of the measured value of the calibration standard can be used as a correction value. Similarly, the Eh probe cannot be calibrated in the field, but is checked against a known standard, such as Zobell solution. The instrument should display a millivolt (mv) value that falls within the

range set by the manufacturer. Because Eh is temperature dependent, the measured value should be corrected for site-specific variance from standard temperature (25 • C). The Eh probe should be replaced if the reading is not within the manufacturer's specified range. All calibration data should be recorded on the Micropurging Groundwater Sampling Data Sheet attached to this SOP or in a logbook.

2.2 WELL PURGING

The well to be sampled should be opened and groundwater in the well allowed to equilibrate to atmospheric pressure. Equilibration should be determined by measuring depth to water below the marked reference on the wellhead (typically the top of the well casing) over two or more 5-minute intervals. Equilibrium conditions exist when the measured depth to water varies by less than 0.01 foot over two consecutive readings. Total depth of well measurement should be made following sample collection, unless the datum is required to place nondedicated sample collection equipment. Depth to water and total well depth measurements should be made in accordance with procedures outlined in SOP No. 014 (Static Water Level, Total Well Depth, and Immiscible Layer Measurement).

If the well does not have a dedicated sample collection device, a new or previously decontaminated portable sample collection device should be placed within the well. The intake of the device should be positioned at the midpoint of the well screen interval. The device should be installed slowly to minimize turbulence within the water in the casing and mixing of stagnant water above the screened interval with water in the screened interval. Following installation, the flow controller should be connected to the sample collection device and the flow-through cell connected to the outlet of the sample collection device. The calibrated groundwater chemistry monitoring probes should be installed in the flow-through cell. If a flow meter is used, it should be installed ahead of the flow-through cell.

If the well has a dedicated sample collection device, the controller for the sample collection device should be connected to the sample collection device. The flow meter and flow-through cell should be connected in line to the discharge tube, and the probes installed in the flow-through cell.

The controller should be activated and groundwater extracted (purged) from the well. The purge rate should be monitored, and should not exceed the capacity of the well. The well capacity is defined as the

maximum discharge rate that can be obtained with less than 0.1 meter (0.3 foot) drawdown. Typically, the discharge rate will be less than 0.5 liters per minute (L/min) (0.13 gallons per minute). The maximum purge rate should not exceed 1 L/min (0.25 gallons per minute), and should be adjusted to achieve minimal drawdown.

Water levels, effluent chemistry, and effluent flow rate should be continuously monitored while purging the well. Purging should continue until the measured chemical and optical parameters are stable. Stable parameters are defined as monitored chemistry values that do not fluctuate by more than the following ranges over three successive readings at 3-minute intervals: ±0.1 pH unit; ±3 percent for SC; ±10 mv for Eh; and ±10 percent for turbidity and DO. Purging will continue until these stabilization criteria have been met or three well casing volumes have been purged. If three casing volumes of water have been purged and the stabilization criteria have not been met, a comment should be made on the data sheet that sample collection began after three well casing volumes were purged. The final pH, SC, Eh, turbidity, and DO values will be recorded. All data should be recorded on the Micropurging Groundwater Sampling Data Sheet attached to this SOP or in a logbook.

2.3 SAMPLE COLLECTION

Following purging, the flow through cell shall be disconnected, and groundwater samples collected directly from the discharge line. Discharge rates should be adjusted so that groundwater is dispensed into the sample container with minimal aeration of the sample. Samples collected for volatile organic compound analysis should be dispensed into the sample container at a flow rate equal to or less than 100 milliliters per minute. Samples should be preserved and handled as described in the investigation field sampling plan or quality assurance project plan.

Tetra Tech EM Inc.

MICROPURGING GROUNDWATER SAMPLING DATA SHEET

Page ____ of ____

Date _____

Well Name _____ Screen Interval _____

Project _____ Station Elevation _____ GND ____ TOC _____ Immiscible Phases Present ☐ Yes ☐ No

Project No. _____ Static Water Level (from TOC) _____ Type _____

Well Location _____ Well Stick Up _____ Measured with _____

Sample Date _____ Static Elevation _____ PID Readings (background) _____

Sampling Personnel _____ Well Depth _____ MEAS ____ RPTD _____ PID Reading (TOC) _____

_____ Feet of Water _____ Wells Installed by _____

Sample ID _____ Gallons/Foot _____ Installation Date _____

Duplicate ID _____ Casing Volume _____ Development Date(s) _____

FIELD CHEMISTRY CALIBRATIONS

Date/Time _____ Spec. Conductance: Standard _____ µmhos/cm at 25• C Reading _____ µmhos/cm at ____• C

pH: pH 4.00 - _____ at _____ • C pH 7.00 - _____ at _____ • C pH 10.00 - _____ at _____ • C Slope _____

Dissolved Oxygen: D.O. Meter _____ mg/L at _____ • C PID: Calibration Gas _____ PPM _____ Span _____ Reading _____

PURGING

Time	Discharge Rate (mL/min)	Dissolved Oxygen (mg/L)	pH	Eh/ORP (mV)	Temp. (•C)	Specific Conduct. (µmhos/cm at •C)	Turbidity (NTU)	Cumulative Volume of Water Removed (Purged)		PID/OVA Reading		Depth to Water (ft)	Comments
								Gallons	Casing Vol.	Location	Value		

SAMPLE PARAMETERS

Condition of well: _____

Remarks: _____

FIELD EQUIPMENT

pH Meter _____ Serial Number _____

Spec. Cond. Meter _____ Serial Number _____

Pump _____ Serial Number _____

Water Level Meter _____ Serial Number _____

D.O. Meter _____ Serial Number _____

Filter Apparatus _____ Filters _____

Temperature Measure _____

Interface Probe _____ Serial Number _____

PID/OVA _____ Serial Number _____

Field Chemistry Calibrations

Fractions _____

_____

_____

Number of Bottles _____

Sample Depth _____

Field Notebook _____

Sample Method _____

_____

Discharge Water Containerized ☐ Yes ☐ No

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GROUNDWATER SAMPLING

SOP NO. 010

REVISION NO. 3

Last Reviewed: March 2000

Quality Assurance Approved

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Date

1.0 BACKGROUND

Groundwater sampling may be required for a variety of reasons, such as examining potable or industrial water supplies, checking for and tracking contaminant plume movement in the vicinity of a land disposal or spill site, Resource Conservation and Recovery Act (RCRA) compliance monitoring, or examining a site where historical information is minimal or non-existent, but where groundwater may be contaminated.

Groundwater is usually sampled through an in-place well, either temporarily or permanently installed. However, it can also be sampled anywhere groundwater is present, such as a pit or a dug or drilled hole.

Occasionally, a well will not be in the preferred location to obtain the sample needed (for example, to track a contaminant plume). In such a case, a temporary or permanent well will have to be installed. An experienced and knowledgeable person, preferably a hydrogeologist, will need to locate the well and supervise its installation so that the samples ultimately collected will be representative of the groundwater. SOP No. 020 (Monitoring Well Installation) provides guidance for installing new monitoring wells.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for determining the quality of groundwater entering, leaving, or affected by site activities through groundwater sampling. The samples are obtained by retrieving water from a well screened in the aquifer(s) underlying a site.

1.2 SCOPE

This SOP provides general guidance for groundwater sampling activities conducted in the field. SOP No. 015 (Groundwater Sample Collection Using Micropurge Technology) provides additional specific guidance for using low flow methods to collect groundwater samples.

1.3 DEFINITIONS

Bailer: A cylindrical sampling device with valves on either end used to extract water from a well. Bailers are usually constructed of an inert material such as stainless steel or polytetrafluoroethylene (Teflon). The bailer is lowered and raised by means of a cable that may be cleaned and reused, or by disposable rope.

Electrical Water Level Indicator: An electrical device that has a light or sound alarm connected to an open circuit used to determine the depth to liquid. The circuit is closed when the probe intersects a conducting liquid. The wire used to raise and lower the probe is usually graduated.

Immiscible Phase: Liquid phases that cannot be uniformly mixed or blended with water. Heavy immiscible phases sink, and light immiscible phases float on water.

Interface Probe: An electrical probe that determines the distance from the surface to air/water, air/immiscible, or immiscible/water interfaces.

Purge Volume: The volume of water that needs to be removed from the well prior to sampling to ensure that the sample collected is representative of the groundwater.

Riser Pipe: The length of well casing above the ground surface.

Total Well Depth: The distance from the ground surface to the bottom of the well.

Water Level: The level of water in a well, measured as depth to water or as elevation of water, relative to a reference mark or datum.

1.4 REFERENCES

U.S. Department of Energy. 1985. "Procedures for the Collection and Preservation of Groundwater and Surface Water Samples and for the Installation of Monitoring Wells: Second Edition." Edited by N. Korte and P. Kearl. Technical Measurements Center, Grand Junction Projects Office. GJ/TMC-08.

U.S. Environmental Protection Agency (EPA). 1977. "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities." EPA-530/SW-611. August.

EPA. 1984. "Sampling at Hazardous Materials Incidents." EPA Hazardous Response Support Division, Cincinnati, 1984.

EPA. 1995. "Groundwater Well Sampling." Environmental Response Team SOP #2007 (Rev. #0.0, 01/26/95). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

U.S. Geological Survey. 1984. "National Handbook of Recommended Methods for Water-Data Acquisition" Reston, Virginia.

1.5 REQUIREMENTS AND RESOURCES

There are various options available to obtain groundwater samples. The procedures are outlined in the following section. The equipment needed to accomplish these procedures includes the following:

- Organic vapor detector with a flame ionization detector (FID) or a photoionization detector (PID)
- Pipe wrench
- Electrical water level indicator or interface probe
- Steel tape with heavy weight
- Purging device (type needed depends on well depth, casing diameter, and type of sample desired; see sampling devices below)
- Sampling device (type needed depends upon depth to water and type of sample desired)
  - Teflon bailer
  - Stainless steel bailer
  - Teflon bladder pump
  - Stainless steel submersible (nonoil-bearing) pump
  - Existing dedicated equipment
  - Peristaltic pump
- Sample containers
- Wastewater containers
- Field logbook
- Stopwatch

Additional equipment is required to complete measurement of field parameters (for example, pH, specific conductance, and temperature) of the groundwater in the well.

2.0 PROCEDURE

Prior to sampling, a site-specific sampling plan should be developed. The plan should take into consideration the site characteristics and should include:

- Specific repeatable well measurement techniques and reference points for determining the depth to water and the depth to the bottom of the well
- Specific method of purging and selection of purging equipment
- Specific methods and equipment for measurements of field parameters
- Specific method of sample collection and the sampling equipment that will be used
- Specific parameters for which samples will be analyzed
- Order in which sample bottles will be filled, based on the analytical parameters

The following sections discuss procedures for approaching the well, establishing a sample preparation area, making preliminary well measurements, purging the well, and collecting samples.

2.1 APPROACHING THE WELL

In general, all wells should be assumed to pose a health and safety risk until field measurements indicate otherwise. Approach wells from the upwind side. Record well appearance and general condition of the protective casing, surface seal, and surrounding area in the logbook.

Once at the well, the lead person should systematically use the organic vapor detector to survey the immediate area around the well (from the breathing zone to the top of the casing to the ground). If elevated FID and PID meter readings are encountered, retreat to a safe area and instruct the sampling team to put on the appropriate level of personal protective equipment (PPE). See SOP No. 003 (Organic Vapor Air Monitoring) for additional guidance.

Upon opening the well casing, the lead person should systematically survey inside the well casing, above the well casing in the breathing zone and the immediate area around the well. If elevated FID or PID meter readings in the breathing zone are encountered (see health and safety plan for action levels), retreat and put on appropriate PPE. It is important to remember that action levels are based on readings in the breathing zone, not within the well casing. Representative organic vapor detector readings should be recorded in the logbook.

2.2 ESTABLISHING A SAMPLE PREPARATION AREA

The sample preparation area is generally located upwind or to either side of the well. If elevated readings are encountered using an organic vapor detector, this area should be taped off and the sample preparation area should be located upwind where ambient readings are found.

2.3 MAKING PRELIMINARY WELL MEASUREMENTS

Several preliminary well measurements should be made prior to initiating sampling of the well. These include determining water level and total well depth measurements, determining the presence of immiscible phases, and calculating purge volumes. All preliminary measurements will be recorded in the logbook as they are determined. SOP No. 014 (Static Water Level, Total Well Depth, and Immiscible Layer Measurement) provides additional information concerning these preliminary measurements.

2.3.1 Water Level and Total Well Depth Measurements

Tetra Tech typically uses an electric water level indicator for water level measurements. This device sounds an alarm or illuminates a light when the measuring probe touches the water surface, thus closing an electrical circuit. The electric cable supporting the probe is usually graduated in feet and can be read at the well site directly. The remaining fraction is measured with a steel tape graduated to 0.01 foot. The distance between the static water level and the marked or notched location at the top of the riser pipe is measured. The height of the riser pipe above ground surface, as obtained from well location survey data, is then subtracted from the total reading to give the depth to static water. To improve accuracy, three separate readings should be made, and the values averaged. This helps to eliminate any errors due to kinks or bends in the cables, which may change in length when the water level indicator is raised and lowered.

The total well depth can be measured by using a steel tape with a heavy weight attached to the end. The tape is lowered into the well until resistance is met, indicating that the weight has reached the bottom of the well. The total well depth is then read directly from the steel tape to the 0.01-foot fraction. The distance between the bottom of the well and the marked or notched location on the riser pipe is measured. The height of the riser pipe above the ground surface, as obtained from well survey data, is then subtracted from the total reading to give the depth to the bottom of the well. To improve accuracy, three separate readings should be made, and the readings averaged.

2.3.2 Determining If Immiscible Phases Are Present

If immiscible phases (organic floaters or sinkers) are present, the following measurement activities should be undertaken. Organic liquids are measured by lowering an interface probe slowly to the surface of the liquid in the well. When the audible alarm sounds, record the depth. If the alarm is continuous, a floating immiscible layer has been detected. To determine the thickness of this layer, continue lowering the probe until the alarm changes to an oscillating signal. The oscillating signal indicates that the probe has detected an aqueous layer. Record this depth as the depth to water and determine the thickness and the volume of the immiscible layer.

Continue lowering the probe into the well to determine if dense immiscible phases (sinkers) are present. If the alarm signal changes from oscillating to a continuous sound, a heavier immiscible layer has been detected; record this depth.

Continue lowering the probe to the bottom of the well and record the total depth. Separate total depth measurements with a steel tape are not necessary when using an interface probe. Calculate and record the sinker phase volume and total water volume in the well. A chart is provided in Table 1 to assist in these calculations. If immiscible phases are present, immediately refer to Section 2.5.3 or 2.5.4 of this SOP.

2.3.3 Determination of Purging Volume

If the presence of floaters or sinkers does not need to be determined, determine the depth to water and the total depth of the well as described in Section 2.3.1. Once these measurements have been made and recorded, use Table 1 to calculate the total volume of water in the well. Multiply this volume by the purging factor to determine purging volume. The minimum purging factor is typically three casing

volumes but may be superseded by site-specific program requirements, individual well yield characteristics, or stabilization of field parameters measured during purging. Field parameters (for example, pH, specific conductance, and temperature) should be measured prior to purging and after each well volume. All field parameter data should be recorded in the field logbook. SOPs No. 011 (Field Measurement of Water Temperature), 012 (Field Measurement of pH), and 013 (Field Measurement of Specific Conductance) include more detailed procedures for determining these field parameters.

In Table 1, the volume of water in a 1-foot section of a 2-inch-diameter well is 0.163 gallon. This chart can easily be used for any water depth by multiplying all the values in Table 1 by the L value (depth, in feet, of water in the well). The volume of water in the well is based on the following formula:

$$V = \frac{\pi \times D^2}{4} \times L$$

where

V = volume of water in the well (cubic feet)
D = inside diameter of the well (feet)
L = depth of water in the well (feet)

2.4 PURGING THE WELL

Currently, Tetra Tech standards allow for six options for purging wells:

1. Teflon bailers
2. Stainless steel bailers
3. Teflon bladder pumps
4. Stainless steel submersible (nonoil-bearing) pumps
5. Existing dedicated equipment
6. Peristaltic pumps (these devices are for shallow wells only)

As previously stated, the minimum purging volume is typically three casing volumes. Exceptions to this standard may be made in the case of low-yield wells. When purging low-yield wells, purge the well once

to dryness. Samples should be collected as soon as the well recovers. When the time required for full recovery exceeds 3 hours, samples should be collected as soon as sufficient groundwater volume is available.

The well should be purged until measured field parameters have stabilized. If any field parameter has not stabilized, additional purging should be performed. To be considered stable, field parameters should change by no more than the tolerance levels listed on Table 2 between each well volume purged.

At no time should the purging rate be high enough to cause the groundwater to cascade back into the well, resulting in excessive aeration and potential stripping of volatile constituents.

The actual volume of purged water can be measured using several acceptable methods:

- When bailers are used, the actual volume of each bailer's contents can be measured using a calibrated bucket.
- If a pump is used for purging, the pump rate can be determined by using a bucket of known volume, stopwatch, and the duration of pumping time necessary to purge the known volume.

2.5 SAMPLE COLLECTION

This section first describes general groundwater sample collection procedures. This section also describes procedures for collecting groundwater samples for volatile organic analysis (VOA) and for collecting samples when light or heavy immiscible layers are present in a monitoring well. Samples of light and heavy immiscible layers should be collected before the well is purged.

2.5.1 General Groundwater Sampling Procedures

The technique used to withdraw a groundwater sample from a well should be selected based on the parameters for which the sample will be analyzed. To ensure that the groundwater samples are representative, it is important to avoid physically altering or chemically contaminating the sample during collection, withdrawal, or containerization. If the samples are to be analyzed for volatile organic compounds, it is critical that air does not become entrained in the water column.

Acceptable sampling devices for all parameters are double check valve stainless steel or Teflon bailers, bladder pumps, low-flow positive displacement pumps, or for shallow wells, peristaltic pumps. Additional measurements of field parameters should be performed at the time of sampling.

In some cases, it may become necessary to use dedicated equipment already in the well to collect samples. This is particularly true of high volume, deep wells (>150 feet) where bladder pumps are ineffective and bailing is impractical. If existing equipment must be used, however, determine the make and model of the pump and obtain information on component construction materials from the manufacturer or facility representatives. If an existing pump is to be used for sampling, make sure the flow volume can be reduced so that a reliable VOA sample can be taken. Record the specific port, tap, or valve from which the sample is collected.

General sampling procedures include the following:

- Clean sampling equipment should not be placed directly on the ground. Use a plastic drop cloth or feed line from clean reels. Never place contaminated lines back on reels.
- Check the operation of the bailer check valve assemblies to confirm free operation.
- If the bailer cable is to be decontaminated and reused, it must be made of Teflon-coated stainless steel.
- Lower sampling equipment slowly into the well to avoid degassing the water and damaging the equipment.
- Pump flow rates should be adjusted to eliminate intermittent or pulsed flow. The settings should be determined during the purging operations.
- A separate sample volume should be collected to measure necessary field parameters. Samples should be collected and containerized in the order of the parameters' volatilization sensitivity. Table 3 lists the preferred collection order for common groundwater parameters.

Intermediate containers should never be used to prepare VOA samples and should be avoided for all parameters in general. All VOA containers should be filled at a single sampling point or from a single bailer volume.

2.5.2 Collection of Volatile Organics Samples

This section discusses the collection of samples for VOA using either a bailer or bladder pump in detail. Other pumps (such as positive displacement or peristaltic) can be used. The following factors are critical to the collection of representative samples for VOA: ensuring that no air has become entrained in the water column, low pump flow rates (less than 100 milliliter [mL] per minute, if possible), and avoiding flow surges.

2.5.2.1 Collection with Bailers

Samples for VOA should be collected from the first bailer removed from the well after purging. The most effective means requires two people. One person should retrieve the bailer from the well and pour its contents into the appropriate number of 40-mL VOA vials held by the second person. Cap each vial and invert it. If a bubble exists, unscrew the cap and add more water, or discard and repeat. The sample should be transferred from the bailer to the sample container in a manner that will limit the amount of agitation in order to reduce the loss of volatile organics from the sample.

Always fill VOA vials from a single bailer volume. If the bailer is refilled, samples cannot be considered duplicates or splits.

2.5.2.2 Collection with a Bladder Pump (Well Wizard)

To successfully perform VOA sampling with a Well Wizard bladder pump, the following steps must be completed:

1. Following manufacturer's directions, activate the pump. Full water flow from the discharge tubing will begin after 5 to 15 pumping cycles. These initial pumping cycles are required to purge air from the pump and discharge tubing. The discharge and recharge settings must be manually set and adjusted to pump at optimum flow rates. To activate the bladder, it is best to set the initial cycle at long discharge and recharge rates.

2. Reduce water flow rate for VOA sample collection. To reduce the water flow rate, turn the throttle control valve (located on the left side of the Well Wizard pump control panel) counterclockwise.

3. Collect VOA sample from discharge tubing. VOA vials must be placed beneath the discharge tubing while avoiding direct contact between the vials and the tubing. Never place tubing past the mouth of the VOA vial. The pump throttle control must be turned as necessary to maintain a trickle of water in order to obtain a meniscus in the vial.

4. Continue with non-VOA sampling. Increase pump flow rate by turning the throttle control knob clockwise.

2.5.3 Collection of Light Immiscible Floaters

The approach used when collecting floaters depends on the depth to the floating layer and the thickness of that layer. If the thickness of the floater is 2 feet or greater, a bottom-filling valve bailer should be used. Slowly lower the bailer until contact is made with the floater surface, and lower the bailer to a depth less than that of the floater/water interface depth as determined by preliminary measurements with the interface probe.

When the thickness of the floating layer is less than 2 feet, and the depth to the surface of the floating layer is less than 15 feet, a peristaltic pump can be used to extract a sample.

When the thickness of the floating layer, however, is less than 2 feet and the depth to the surface of the floating layer is beyond the effective "lift" of a peristaltic pump (greater than 25 feet), a bailer can be modified to allow filling from the top only (an acceptable alternative is to use a top- loading Teflon or stainless-steel bailer). Disassemble the bailer's bottom check valve and insert a piece of 2-inch diameter Teflon sheet between the ball and ball seat. This will seal off the bottom valve. Remove the ball from the top check valve, thus allowing the sample to enter from the top. To overcome buoyancy when the bailer is lowered into the floater, place a length of one-inch stainless steel pipe on the retrieval line above the bailer (this pipe may have to be notched to allow sample entry if the pipe remains within the top of the bailer). As an alternative, use a top-loading stainless-steel bailer. Lower the device, carefully measuring the depth to the surface of the floating layer, until the top of the bailer is level with the top of the floating layer. Lower the bailer an additional one-half thickness of the floating layer and collect the sample. This technique is the most effective method of collection if the floating layer is only a few inches thick.

2.5.4 Collection of Heavy Immiscible Sinkers

The best method for collection of sinkers is use of a double check valve bailer. The key to collection is controlled, slow lowering and raising of the bailer to and from the bottom of the well. Collection methods are equivalent to those described in Section 2.5.3 above.

TABLE 1

LIQUID VOLUME IN A 1-FOOT SECTION OF WELL CASING

Well Casing Inside Diameter (D) (inches)	Volume of Liquid in 1-Foot Well Section (gallons) $V = 0.0408 (D^2)$
1	0.041
1.5	0.092
2	0.163
3	0.367
4	0.653

TABLE 2

FIELD MEASUREMENT TOLERANCE LEVELS

Field Parameter	Tolerance Level
pH	0.1 pH unit
Specific Conductance	10 percent relative percent difference (RPD)[a]
Temperature	1 °C

Note:

[a] RPD can be determined as follows:

$$RPD = \frac{(\text{Measurement 1} - \text{Measurement 2}) \times 100}{(\text{Measurement 1} + \text{Measurement 2}) / 2}$$

TABLE 3

ORDER OF PREFERRED SAMPLE COLLECTION

1. VOA
2. Purgeable organic halogens (POX)
3. Total organic halogens (TOX)
4. Cyanide
5. Extractable organics
6. Purgeable organic carbon (POC)
7. Total metals
8. Dissolved metals
9. Total organic carbon (TOC)
10. Phenols
11. Sulfate and chloride
12. Nitrate and ammonia
13. Radionuclides

Guidelines for the Measurement of Water Levels in Wells

The accurate and precise determination of ground water gradients and temporal variations of ground water levels requires the monitoring of ground water levels with a standard approach, well maintained equipment and careful, thorough record keeping. The following guidelines describe the methods to be used for the monitoring of ground water levels in monitoring wells.

Water levels measured during drilling are not considered as part of the water level database and should be recorded separately from post well-construction water level data. During drilling, water levels are normally taken relative to ground surface, and recorded in the drilling log, and/or water sampling logbook if relevant. Once the well has been developed and surface construction completed, water levels can be measured relative to an accurately surveyed reference point. Water levels measured after establishment of the reference point are considered as part of the water level database for a site and the following procedures have been prepared to ensure consistency of measurement method and minimization of random measurement error.

1. Establishment of a Water Level Monitoring Point

1.1 Establish and identify a measurement reference point. The preferred place for this is the top of the well casing. Make a permanent mark at the measurement point. A physical mark such as a saw or knife cut in addition to marker pen drawn arrow is recommended.

1.2 Clearly mark the well identification number or name of the measurement location near the measurement reference point.

1.3. Record well identification details in the water level logbook an example of which is provided in Appendix 1. Make sure that the measuring reference point position is noted precisely.

1.4. Measure and record the depth to the ground water in the well as described below in section 2.

1.5. Measure and record the depth of the well using a weighted tape. Lower the tape in the well until it goes slack. Record the depth of the well below ground surface and confirm that the well is clear across the screen interval by comparing the measured depth with the well construction log.

2. Water Level Measurement by Hand

Use a graduated electronic water sensor measuring tape (water level meter) for the measurement of water levels by hand. The depth intervals on the tape should be calibrated periodically using an accurate measuring device. The frequency of calibration depends on the amount of use and if the cable is believed to have been stretched for any reason. At a minimum the calibration should be checked at the beginning of a new project or sampling program. The steps for the hand measurement of water levels are as follows:

2.1. Check for proper operation of the water level meter by immersing the sensor tip in water. Clean and dry the sensor tip before lowering it into a borehole.

2.2. Measure the water level by slowly lowering the cable, until the sensor indicates contact with the water.

2.3. Fine tune the measurement by raising and lowering the cable in gradually smaller increments until the most precise reading is obtained.

2.4 . Read the depth to the water from the water level meter at the measurement reference point and record the value in the logbook. Include the date and time of the reading and note the prevailing weather conditions.

2.5. Compare the water level with any previous data from that site. If the water level measurement is inconsistent with previous values, take a second reading to check for gross measurement reading errors before removing the water level measuring device .

2.6. Decontaminate the portion of the tape and sensor that was immersed before moving to the next well. Spray the cable and sensor with DI water and dry the tip with a disposable paper towel.

3. Automatically Recorded Water Levels

Accurate measurement of water levels using pressure transducers and automatic dataloggers increases the need for organized, thorough and detailed record keeping. Each pressure transducer-datalogger system requires a specific set of protocols to ensure good system operation. All datalogging systems should be installed under a more generic Standard Operating Procedure as follows:

Test Setup

3.1. Measure and record the water level and the depth of the well using the procedures described in section 2.

3.2 Select a depth of installation for the transducer.

The installation depth must be within the specified transducer operating range and allow for the maximum anticipated range of water level variation in the borehole. ***Do not immerse the transducer to more than its operating depth, permanent damage can result.***

3.3. Place a mark on the transducer cable at the selected installation depth.

The transducer cable may already be marked at five or ten feet intervals. If so choose select one of these marks as the transducer installation depth.

3.4 Lower the transducer into the borehole to the required installation depth and attach it securely to the well (example methods are shown in Figures 1 through 5).

When attaching the transducer avoid sharp edges that might kink or tear the cable. ***Do not bend the cable with less than 1-inch radius curves or obstruction of the vent tube in the cable may result.*** *The wellhead and datalogger should be protected against disturbance or damage due to; severe weather, vandalism, curious locals, and traffic.*

3.5. Connect the transducer to the datalogger.

3.6 Enter the required setup parameters for the transducer, calibration information and the details of the desired monitoring schedule (see equipment manuals for the individual datalogging systems).

- ***Be sure to check the date and time setting of the datalogger internal clock.***
- *When operating more than one datalogger at a site be sure to synchronize all internal datalogger clocks to local time.*
- *When more than one channel is connected to a datalogger, record and double check which transducer is measuring which borehole, through which channel.*

3.7. Check the transducer calibration by raising the transducer cable a known amount (the larger the better) and taking a new transducer reading (it is normally possible to interrogate the datalogger for a transducer reading without starting a test).

The recorded water level change should agree with the change in transducer height to within 1 % or 0.1 ft. per 10 ft of water level change.

- *If the error exceeds this amount check all calibration settings. If the degree of error is confirmed, test the calibration again by raising the transducer a known distance. Allow extra time for water levels to re-equilibrate to the change in depth of the transducer.*
- *If the calibration is accurate return the transducer to its reference position.*

3.8. After completion of the calibration test and installation of the transducer at its reference position, allow water levels to fully re-equilibrate before moving to the next step.

3.9. Hand measure and record the water level again as accurately and precisely as possible.

3.10. Set the datalogger reference water level reading to equal the hand water level just measured.

3.11. Start the datalogger test.

A one-hour measurement interval with measurements taken on the hour is the standard for long term water level monitoring (one week or longer). Shorter intervals are generally only used to provide detailed monitoring of the effects of well pumping.

3.12. Confirm that the test has started and that data is being successfully collected by the datalogger.

3.13. Before leaving the site, check that all datalogger connectors and weatherproof caps are properly in place. Close and secure the well cover and any datalogger enclosure.

Data Downloading and Test Completion

3.14. When returning to the site check for signs of any damage or disturbance of the well or datalogger since the previous reading. In particular, check that the transducer is still set at its original reference position.

3.15 Take a current transducer reading by interrogating the transducer directly via the datalogger .

3.16 Take a hand water level reading (section 2) as soon as possible after step 3.15. and record the reading in the logbook.

The transducer reading should be taken before the hand water level measurement to ensure that the transducer reading is not affected by immersion of the water level meter.

3.17. Compare the change in water levels in the logbook for both the pressure transducer and hand measurements since the previous readings.

When the automatic water level recorders are functioning properly, the transducer and hand readings should agree to within 0.05 ft. If the difference is greater than this, check the water level again for both the transducer and hand measurements. If the difference is confirmed the calibration of the transducer should be checked as in step 3.7. If the calibration is still accurate, return the transducer to its previous reference position for continued data

recording or the start of a new test. The sources of such non-transducer calibration error could be;

- *severe temperature changes (check weather records for this possibility),*
- *previous inaccurate hand water level measurements (accurate readings in the future should eliminate this error), or*
- *inaccurate hand water level meter (check the calibration of the meter against a reliable measure).*

3.18. Download the data for the current test before stopping a test. The following convention is used for naming downloaded data files:

Make a new sub directory using the site name (i.e. Bruno) then an eight letter code consisting of:

- site name (two letters),
- well ID (two numbers),
- date of download (two letters for the month and two numbers for the date),
- then a three letter suffix (.DAT for asci files, .BIN for binary files).

An example file name might be BR01MR20.DAT for the March 20 download from SB01 at Bruno.

3.19. Once the download is complete, convert the datafile into a format suitable (e.g. EXCEL spreadsheet) for quick scanning of the data. Scan the data to check that the full data record is in the file and that the equipment appears to be functioning properly.

3.20. Stop or continue the current test as required.

Starting a new test reduces the size of file to be downloaded the next time. It also allows for increasing the available datalogger memory. ***Never delete files from the datalogger until downloaded data has been checked and backed up onto floppy disk.***

3.21. Before leaving the site, check that all datalogger connectors and weatherproof caps are properly in place. Close and secure the well cover and any datalogger enclosure.

3.22. At the end of the day make a disk backup of all downloaded data.

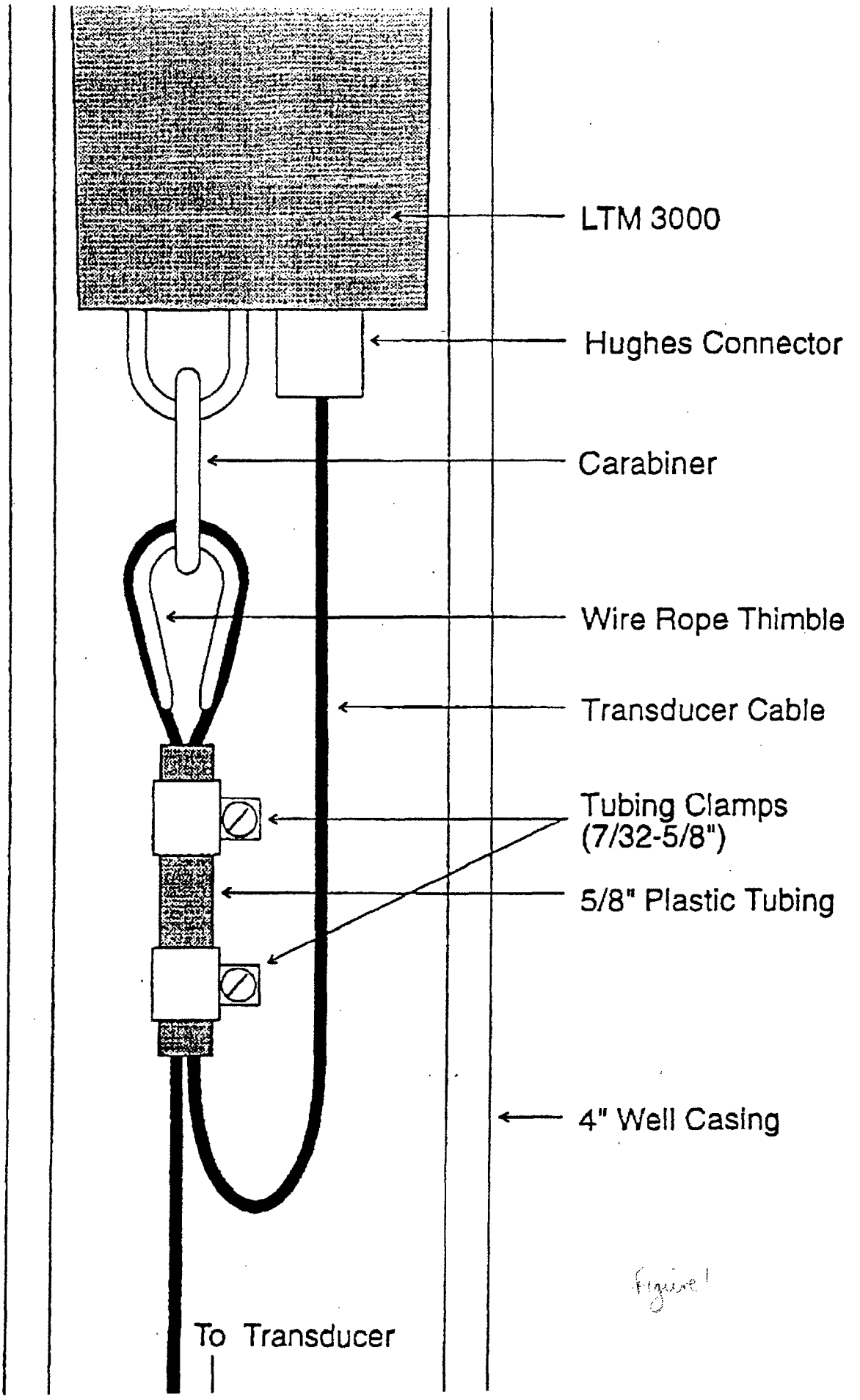

LTM 3000
Hughes Connector
Carabiner
Wire Rope Thimble
Transducer Cable
Tubing Clamps
(7/32-5/8")
5/8" Plastic Tubing
4" Well Casing
To Transducer

Figure 1

Transducer Installation Using Kellems Grip

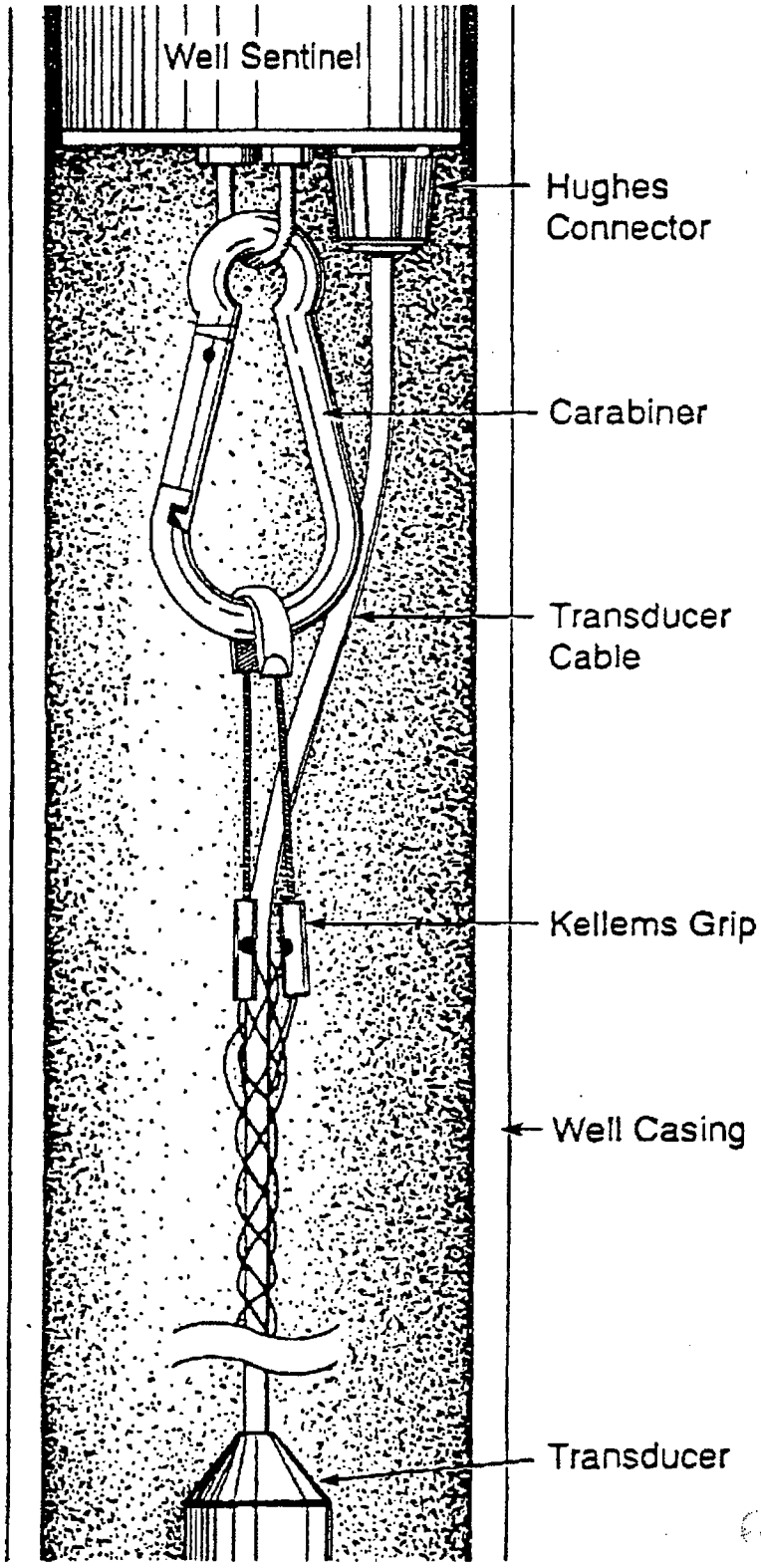
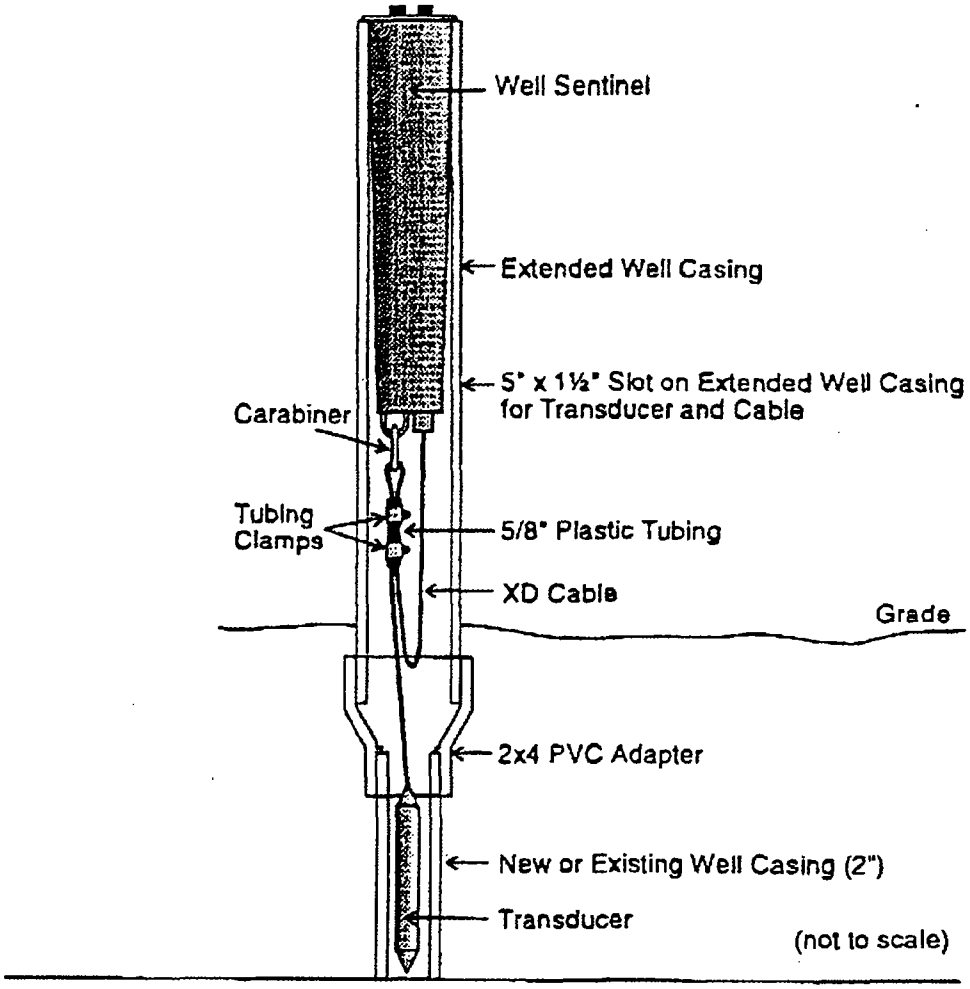


Figure 2


Well Sentinel
Extended Well Casing
5" x 1½" Slot on Extended Well Casing for Transducer and Cable
Carabiner
Tubing Clamps
5/8" Plastic Tubing
XD Cable
Grade
2x4 PVC Adapter
New or Existing Well Casing (2")
Transducer
(not to scale)

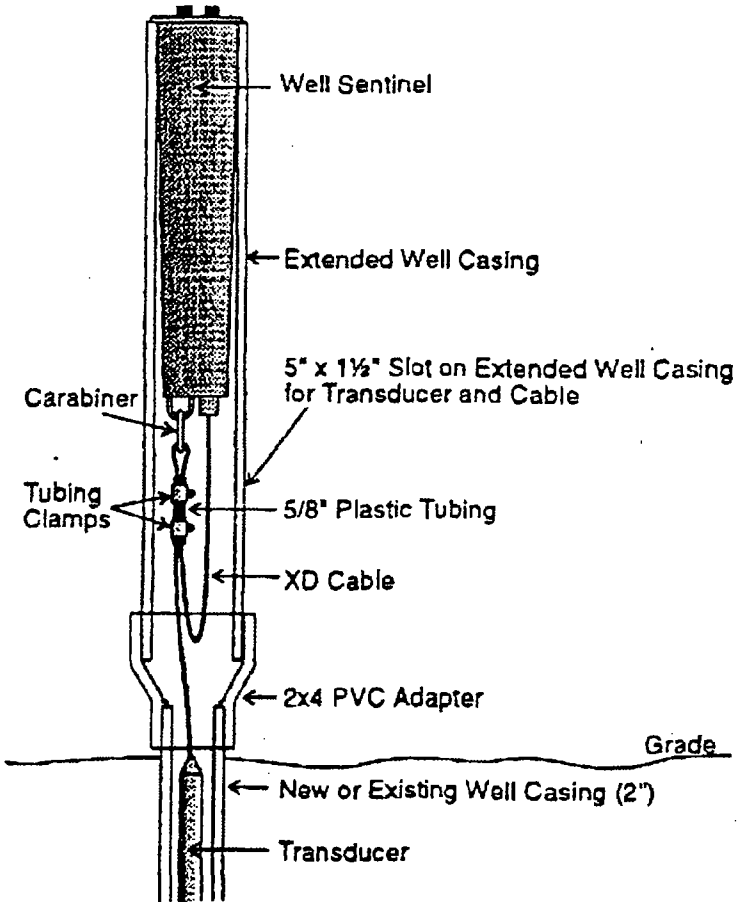
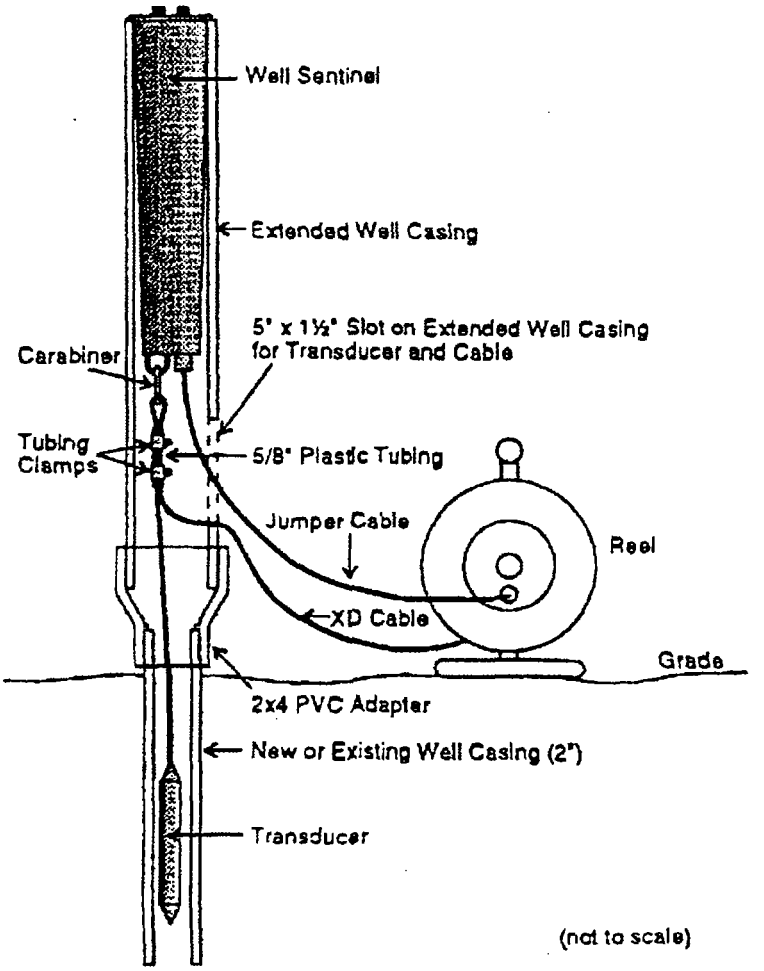

Well Sentinel
Extended Well Casing
5" x 1½" Slot on Extended Well Casing for Transducer and Cable
Carabiner
Tubing Clamps
5/8" Plastic Tubing
XD Cable
2x4 PVC Adapter
Grade
New or Existing Well Casing (2")
Transducer

Figure 3


Well Sentinel
Extended Well Casing
5" x 1½" Slot on Extended Well Casing for Transducer and Cable
Carabiner
Tubing Clamps
5/8" Plastic Tubing
Jumper Cable
Reel
XD Cable
Grade
2x4 PVC Adapter
New or Existing Well Casing (2")
Transducer
(not to scale)

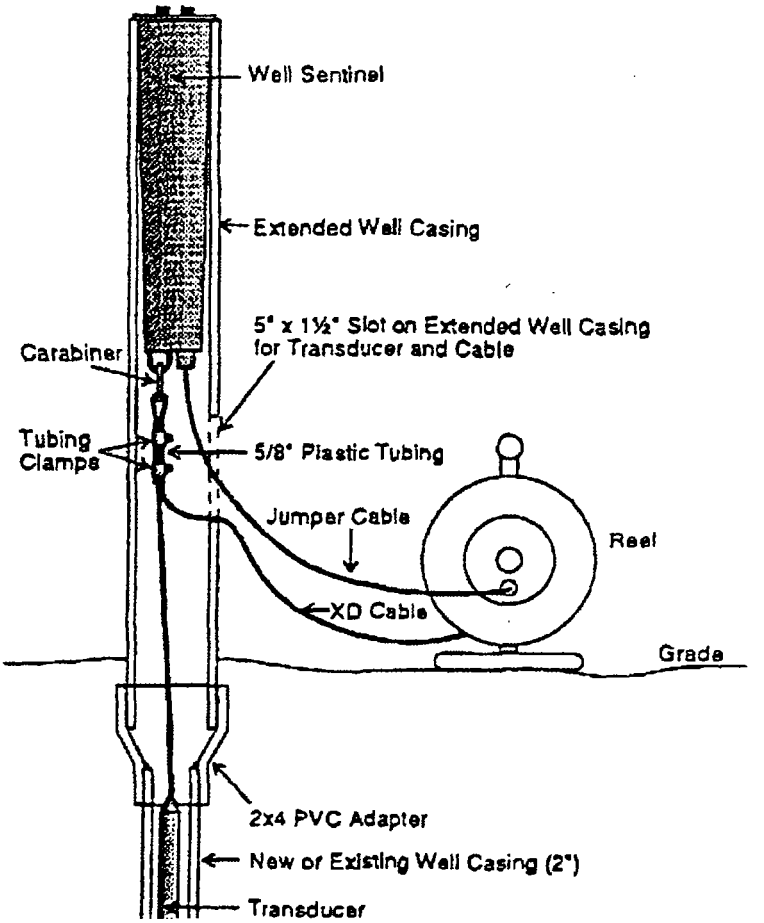

Well Sentinel
Extended Well Casing
5" x 1½" Slot on Extended Well Casing for Transducer and Cable
Carabiner
Tubing Clamps
5/8" Plastic Tubing
Jumper Cable
Reel
XD Cable
Grade
2x4 PVC Adapter
New or Existing Well Casing (2")
Transducer

Figure 4

LTM 3000 Installation at Limited Access Well

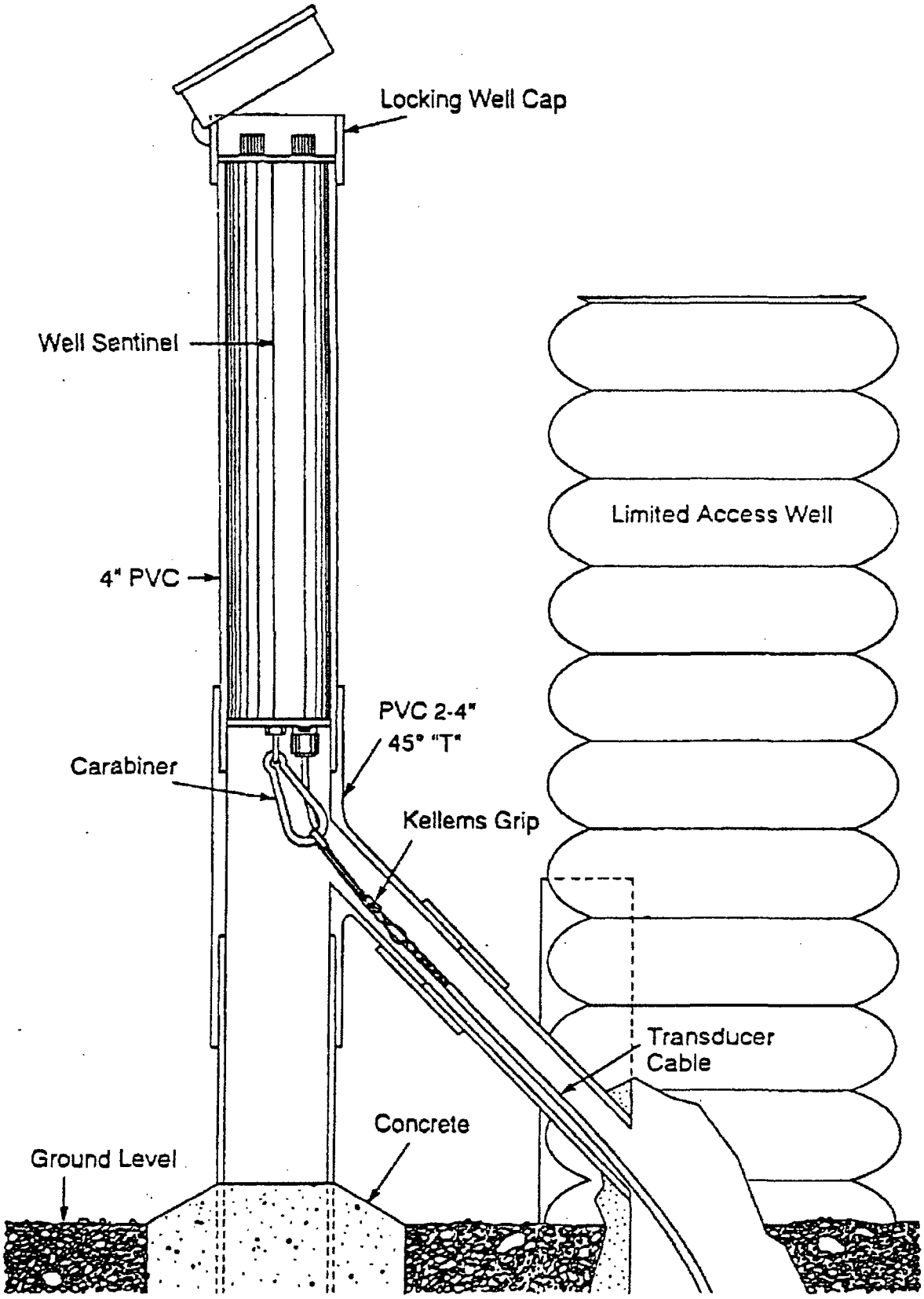


Figure 5

WATER LEVEL LOGBOOK

Site:_____

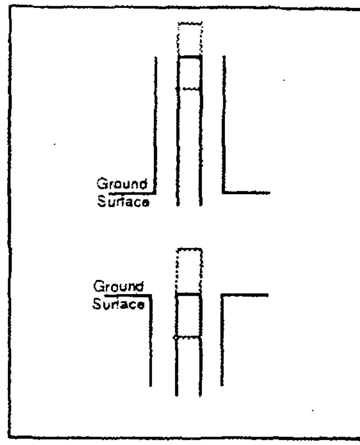
#_____

ANL-AGEM WATER LEVEL LOG SHEET

Site: _____ Well ID: _____ Location: _____

Sketch Map of Location

Date/TIme	Comments	Initials



Site: ____________________

Well # or ID: ____________________

Aquifer Description: ____________________

Well Coordinates X____________ Y____________

Surveyed Elev ________(ft) Well Depth _____(ft below ref)

Height of ref point above ground _____(ft)

Screen Depth: ______ to ______ ____ (ft below surface)

Type of inner casing: ____________________

Indicate the position of reference pointon riser pipe diagram (right)

				Datalogger values		
Date	Time	Hand WL (ft below M.P.)	Weather (temp, description)	Transducer (TX) reading (head or ΔWL)	TX WL (ft below ref.)	Initials

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SURFACE WATER SAMPLING

SOP NO. 009

REVISION NO. 3

Last Reviewed: December 1999

Quality Assurance Approved

May 19, 1993

Date

1.0 BACKGROUND

Surface water sampling is conducted to determine the quality of surface water entering, leaving, or affected by a site. Surface water bodies that can be sampled include streams, rivers, lakes, ponds, lagoons, and surface impoundments. This standard operating procedure (SOP) discusses common methods of collecting grab samples that represent water quality in a water body at a particular point in time.

A series of grab samples also can be composited to represent water quality over a longer period of time. Composite samples can be flow proportional or time proportional. The details of compositing water samples are not included in this SOP.

1.1 PURPOSE

This SOP establishes the requirements and procedures for surface water sampling.

1.2 SCOPE

This SOP applies to surface water sampling and the instruments and methods used to collect the samples.

1.3 DEFINITIONS

Kemmerer Sampler: A messenger-activated water sampling device. Water flows through the device until the release mechanism is triggered to close the container.

Peristaltic Pump: A rotary, positive-displacement pumping device characterized by its low suction and rhythmic nature, and by the fact that the pump does not come into direct contact with the water being sampled.

Pond Sampler: A sampling device fabricated by using an adjustable beaker clamp to attach a beaker to a telescoping, heavy-duty aluminum pole.

1.4 REFERENCES

deVera, E.R., and others. 1980. "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA-600/2-80-018. January.

U.S. Environmental Protection Agency (EPA). 1977. "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities." EPA-530/SW-611. August.

EPA. 1984. "Characterization of Hazardous Waste Sites — A Methods Manual, Volume II. Available Sampling Methods." Second Edition. EPA-600/4-84-076. December.

EPA. 1994. "Surface Water Sampling." Environmental Response Team SOP #2013 (Rev. #0.0, 11/17/94). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

Surface water sampling requires a variety of procedures and instruments. The choice of procedure should be determined by site-specific conditions, such as the type of surface water body, the sampling depth, and the sample location's distance from shore.

Samples can be collected from shallow depths by submerging the sample container. An intermediary disposable collection container or one constructed of a nonreactive material also may be used. A pond sampler, a peristaltic pump, or a Kemmerer sampler may be used to provide extended reach. The following equipment may be required to sample surface water:

- Decontamination materials
- Sample containers and labels
- Point-source bailer
- Dipper
- Boat
- Pond sampler
- Peristaltic pump with batteries or power source
- Silicone tubing
- Heavy-wall Teflon® tubing
- Kemmerer sampler
- Logbook or field sheets

- Chain-of-custody documentation
- Shipping materials

2.0 PROCEDURES

Safe access, handling, and other physical limitations should be influential factors during surface water sampling. A site-specific sampling plan should delineate which of the procedures described below will be used. Any deviations from the sampling plan should be recorded in the site-specific field logbook.

The following subsections provide detailed procedures for surface water sampling using specific instruments and methods. In all cases, select a sampling location where the water quality will best represent the water chemistry of the water body. Avoid stagnant or fast-moving areas. Do not sample immediately downstream of incoming tributaries, because of the likelihood of incomplete mixing.

2.1 SURFACE WATER SAMPLING BY SUBMERGING SAMPLE CONTAINER

Samples from shallow depths should be collected by submerging the sample container. This method is advantageous when the sample might be significantly altered during transfer from a collection vessel into another container. This method should not be used for sampling lagoons or surface impoundments where contact with contaminants is a potential concern.

The following procedure can be used for sampling surface water by submerging the sample container:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017 (Sample Collection Container Requirements).

2. If required by the project, measure the temperature, pH, and specific conductance of the surface water body before collecting the sample using procedures in SOPs No. 11 (Field Measurement of Water Temperature), No. 12 (Field Measurement of pH in Water), and No. 13 (Field Measurement of Specific Conductance), respectively. Record this information on the field sheet or in the logbook.

3. For stream sampling, sample the location farthest downstream first. Orient the mouth of the sample container upstream while standing downstream so as not to stir up any sediment that would contaminate the sample.

4. For a larger body of surface water, such as a lake, collect samples near the shore, unless boats are feasible and permitted. Collect samples from shallow depths by submerging the sample container.

5. Collect surface water samples at each location before collecting sediment samples to avoid contaminating the water samples with excess suspended particles generated during sediment sampling.

6. Continue delivery of the sample until the container is almost full. If sampling for volatile organic compounds (VOC), the container must be completely filled leaving no head space.

7. Preserve the sample in accordance with requirements in SOP No. 16 (Sample Preservation and Maximum Holding Times). Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

8. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

2.2 SURFACE WATER SAMPLING WITH TRANSFER DEVICE

A dipper, bailer, or other device made of inert material, such as stainless steel or Teflon®, can be used to transfer liquid samples from their source to a sample container. This prevents contamination of the outside of the sample container as a result of direct immersion in surface water. Depending on the sampling application, the transfer device may be either disposed of or reused. If reused, the device should be thoroughly rinsed and decontaminated prior to sampling a different source.

A transfer device can be used in most sampling situations. However, direct collection by submerging the sample container is the preferred method when (1) aeration of the sample must be avoided (as in sampling surface water for VOCs) or (2) a significant amount of the sample may be lost due to adhesion to the transfer device.

The following procedure can be used for sampling surface water with a dipper, bailer, or other transfer device:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017 (Sample Collection Container Requirements).

2. If required by the project, measure the temperature, pH, and specific conductance of the surface water body before collecting the sample using procedures in SOPs No. 11 (Field Measurement of Water Temperature), No. 12 (Field Measurement of pH in Water), and No. 13 (Field Measurement of Specific Conductance), respectively. Record this information on the field sheet or in the logbook.

3. With minimal surface water disturbance, submerge a precleaned dipper, bailer, or other transfer device.

4. Allow the device to fill slowly and continuously.

5. Retrieve the device from the surface water with minimal disturbance.

6. Remove the cap from the sample container. Slightly tilt the mouth of the container below the edge of the transfer device.

7. Empty the device slowly, allowing the sample to flow gently down the inside of the container with minimal entry turbulence. Continue delivery of the sample until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

8. Preserve the sample in accordance with requirements in SOP No. 16 (Sample Preservation and Maximum Holding Times). Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

10. Decontaminate the transfer device prior to reuse or storage using the procedures in SOP No. 002, General Equipment Decontamination.

2.3 SURFACE WATER SAMPLING WITH POND SAMPLER

A pond sampler may be used to collect liquid samples from ponds, pits, and lagoons (see Figure 1). A pond sampler is easily and inexpensively fabricated. To construct a pond sampler, use an adjustable clamp to attach a sampling beaker to the end of a two- or three-piece telescoping aluminum tube. The telescoping tube serves as the handle. Nondisposable equipment should be cleaned before and after each use.

The following procedure can be used for sampling surface water with a pond sampler:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017 (Sample Collection Container Requirements).

2. If required by the project, measure the temperature, pH, and specific conductance of the surface water body before collecting the sample using procedures in SOPs No. 11 (Field Measurement of Water Temperature), No. 12 (Field Measurement of pH in Water), and No. 13 (Field Measurement of Specific Conductance), respectively. Record this information on the field sheet or in the logbook.

3. Assemble the pond sampler. Ensure that the sampling beaker, bolts, and nuts securing the clamp to the pole are tightened properly.

4. Collect the sample by slowly submerging the precleaned beaker with minimal surface water disturbance.

5. Retrieve the pond sampler from the surface water with minimal disturbance.

6. Remove the cap from the sample container. Slightly tilt the mouth of the container below the edge of the beaker.

7. Empty the beaker slowly, allowing the sample to flow gently down the inside of the container with minimal entry turbulence. Continue delivery until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

8. Preserve the sample in accordance with requirements in SOP No. 16 (Sample Preservation and Maximum Holding Times). Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

9. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

10. Decontaminate the pond sampler prior to reuse or storage using the procedures in SOP No. 002, General Equipment Decontamination.

2.4 SURFACE WATER SAMPLING WITH PERISTALTIC PUMP

To extend reach in sampling efforts, a small peristaltic pump can be used (see Figure 2). A peristaltic pump draws the sample through heavy-wall Teflon® tubing and pumps it directly into the sample container. Use of a peristaltic pump allows the operator to reach out into a liquid body, to sample from a depth or to sweep the width of a narrow stream. A battery-powered pump is preferable because it eliminates the need for a direct current generator or an alternating current inverter.

If medical-grade silicone tubing is used in the peristaltic pump, it is suitable for sampling almost any parameter, including most organics. However, some VOC stripping may occur and some sample material

may adhere to the tubing. Teflon® tubing may be used in place of silicon tubing on the intake side of the pump to minimize the amount of sample adherence to the tubing. If tubing is to be reused, it should be cleaned before and after each use. Depending on project requirements, it may be necessary to replace the Teflon® intake tubing and the pump silicon tubing between sampling locations to prevent cross contamination.

Procedures for sampling surface water with a peristaltic pump are summarized below:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017 (Sample Collection Container Requirements).

2. If required by the project, measure the temperature, pH, and specific conductance of the surface water body before collecting the sample using procedures in SOPs No. 11 (Field Measurement of Water Temperature), No. 12 (Field Measurement of pH in Water), and No. 13 (Field Measurement of Specific Conductance), respectively. Record this information on the field sheet or in the logbook.

3. Install clean, medical-grade silicone tubing in the pump head according to the manufacturer's instructions. Allow enough tubing on the discharge side to facilitate delivery of liquid into the sample container. Allow only enough tubing on the suction end for attachment to the intake line. This will minimize sample contact with the tubing.

4. Select the length of intake tubing needed to reach the required sample location. Attach it to the intake side of the pump tubing. Heavy-wall Teflon® tubing of a diameter equal to that of the required pump tubing suits most applications. A heavier tubing wall will allow slightly greater lateral reach.

5. If possible, allow several liters of surface water to pass through the pump before collecting the sample. Collect this purge volume. Return it to the source after the samples have been withdrawn.

6. Fill the sample container by allowing the pump discharge to flow gently down the inside of the bottle with minimal entry turbulence. Continue delivery of the sample until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

7. Preserve the sample in accordance with requirements in SOP No. 16 (Sample Preservation and Maximum Holding Times). Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

8. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

9. Allow the pump to drain, and then disassemble it. Decontaminate the tubing before reuse using the procedures in SOP No. 002 (General Equipment Decontamination) or dispose of it.

2.5 SURFACE WATER SAMPLING WITH KEMMERER SAMPLER

The Kemmerer sampler (see Figure 3) is used to collect surface water samples when the required sample depth is greater than that which can be sampled with a pump. A Kemmerer sampler may be constructed of various materials to be compatible with the required analytical technique. The sampler should be cleaned before and after each use.

Procedures for sampling surface water with a Kemmerer sampler are summarized below:

1. Place all equipment on plastic sheeting next to the sampling location. Sample containers should be selected in accordance with the requirements in SOP No. 017 (Sample Collection Container Requirements).

2. If required by the project, measure the temperature, pH, and specific conductance of the surface water body before collecting the sample using procedures in SOPs No. 11 (Field Measurement of Water Temperature), No. 12 (Field Measurement of pH in Water), and No. 13 (Field Measurement of Specific Conductance), respectively. Record this information on the field sheet or in the logbook.

3. Inspect the body of the Kemmerer sampler to ensure that the drain line valve is closed, as appropriate. Measure and mark the sample line (cable) at the desired sampling depth.

4. Open the sampler by lifting the upper stopper-trip head assembly.

5. Gradually lower the sampler into the surface water until the sample liquid reaches the sample line.

6. Place a messenger on the sample line and release it, closing the sampler.

7. Retrieve the sampler. Prevent accidental opening of the lower stopper by holding the center rod of the sampler.

8. Rinse or wipe off the exterior of the sampler. Recover the sample by grasping the lower stopper and sampler body with one hand. Transfer the sample by lifting the upper stopper with the other hand and carefully pouring the contents into the sample container. If a drain line valve is present, hold the valve over the sample container, and open the valve slowly to release the sample.

9. Transfer the sample slowly, allowing it to flow gently down the inside of the container with minimal entry turbulence. Continue delivery until the container is almost full. If sampling for VOCs, the container must be completely filled leaving no head space.

10. Preserve the sample in accordance with requirements in SOP No. 16 (Sample Preservation and Maximum Holding Times). Ensure that a Teflon® liner is present in the cap of the sample container if required. Secure the cap tightly and affix a completed sample label to the container.

11. Complete all chain-of-custody documentation, field logbook entries, and sample packaging requirements.

12. Decontaminate the Kemmerer sampler prior to reuse or storage using the procedures in SOP No. 002, General Equipment Decontamination.

FIGURE 1

POND SAMPLER

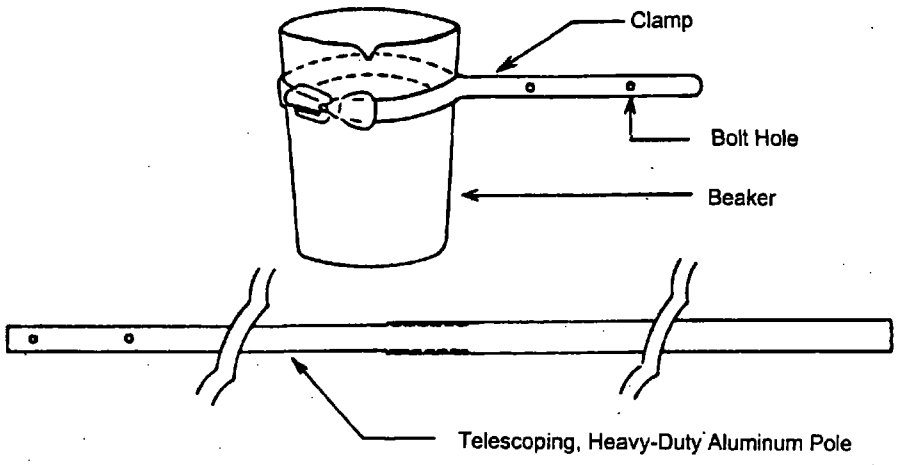


FIGURE 2

PERISTALTIC PUMP FOR LIQUID SAMPLING

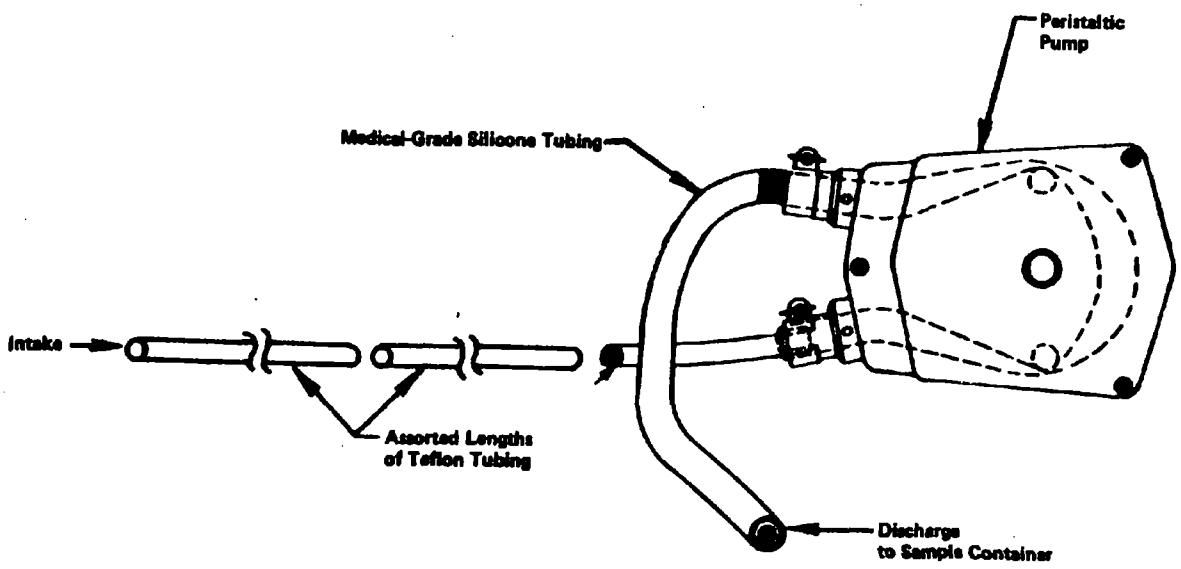
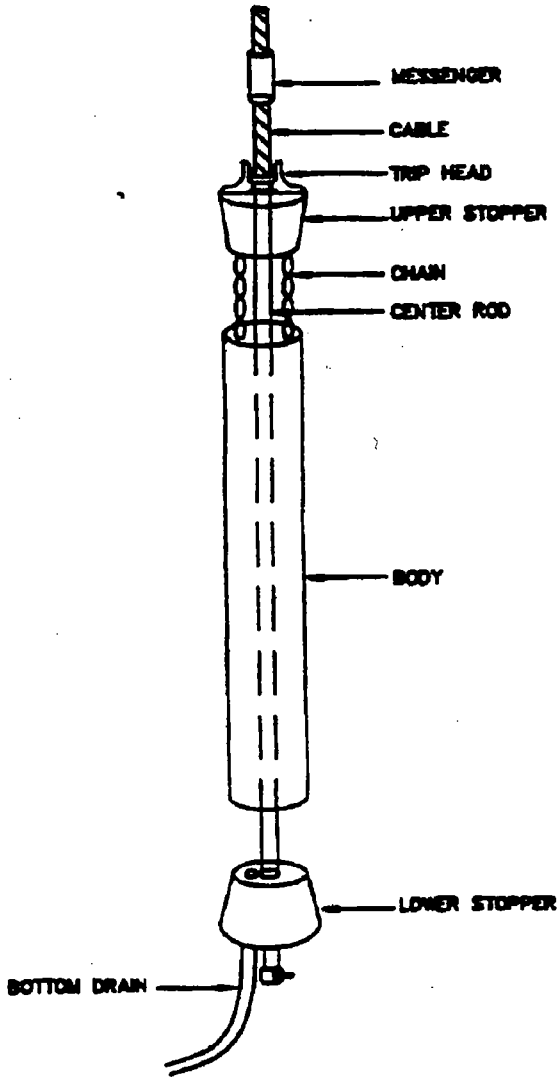


FIGURE 3

KEMMERER SAMPLER



COLLECTING WATER-QUALITY SAMPLES FOR DISSOLVED METALS-IN-WATER

U.S. Environmental Protection Agency, Region 6
Compiled by Forrest B. John

Revised January 13, 2000

COLLECTING WATER-QUALITY SAMPLES FOR DISSOLVED METALS-IN-WATER

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Section 1 Preparation of Field and Laboratory Equipment

Cleaning equipment (i.e. sample bottles, pump tubing, etc.) to be used for clean chemistry applications, both in the field and in the laboratory, is a critically important procedure. Improperly prepared equipment can result in a wasted sampling or analysis efforts and the need for re-sampling and re-analysis. Both problems can result in significant project cost over-runs. It is very important that the procedures described in this SOP be followed carefully and completely at all times.

1.1 Apparatus and Materials

As a minimum, all cleaning should be done in a laboratory reserved for low-level metals work. To the greatest extent possible, the final rinses of each piece of equipment should be done in a clean bench or clean room.

4. High density polyethylene (HDPE) or polypropylene (PP) tubs (preferably with covers) to be used for detergent washing of all equipment and acid soaking of larger items.
5. Wide mouth HDPE or PP jars or carboys for acid soaking smaller items.
6. Various sizes of larger, clear low density polyethylene (LDPE) plastic bags, kept dust free, for covering equipment at various stages during the cleaning process.
7. Sample bottles (LDPE or teflon) of various sizes (i.e. 30 ml to 8 liter).
8. Teflon™ (FEP) tubing.
9. C-flex brand peristaltic pump tubing.
10. Various sizes of plastic cable ties to seal bags at various stages during the cleaning process.
11. Dust-free (e.g. tyvek type) laboratory coats and hair covers to be worn during all stages of the cleaning process.
12. Powder-free gloves to be worn during all stages of the cleaning process.

1.2 Reagents

1. Acids used in the cleaning steps must be ACS reagent grade or better. The ability to achieve acceptable bottle and sampler blanks (i.e. all elements below the detection limit required by the data quality objectives) should be the determining factor for the grade of acid used.
   - ✓ Hydrochloric acid (~10%): Add 270 ml concentrated HCl to 400 ml Type II water and dilute to 1 liter
   - ✓ Nitric acid (1:1): Add 500 ml concentrated HNO_3 to 400 ml Type II water and dilute to 1 liter.
2. Reagent water with metal levels not detectable by ICP-MS. Water should be monitored for impurities. Sources of ultra-pure water, including distilled-deionized, sub-boiling quartz distilled, and Milli-Q, should be compared periodically to select the cleanest water for calibration standard and sample preparation.
3. Ultrapure ammonium hydroxide redistilled from ACS reagent grade material.

1.3 Equipment Cleaning Procedures

1.3.1 Teflon™ Tubing

1. If the tubing has been used previously, cut a small piece of Handy-wipe to fit snugly in tubing.
2. Wipe air valve by DIW container off to get any loose debris off.
3. Attach tubing to air valve and turn on with just enough air pressure to push the Handy-wipe through slowly.
4. Repeat this step 3 times.
5. Clean the outside of the tubing with micro cleaner and designated clean scrubber. Cable tie each set of tubing together, and attach a small piece of clean No. 24 c-flex tubing to each tubing set.
6. Carefully remove tubing that is feeding DIW (distilled water) container.
7. Attached tubing with a cable tie and cover tubing with a plastic bag.
8. Cut a small hole in the bottom of the bag to have access to the tubing.
9. Attach a small piece of clean No. 24 c-flex tubing on tubing set to DIW tubing and flush at appropriate feeding rate.
10. After tubing set has been flushed out thoroughly, empty DIW out of tubing and place in clean bag. Label bag as to stage of cleaning.
11. Once all tubing sets have been flushed, emptied and bagged, return DIW tubing to the container and cover it with plastic.
12. Using designated micro solution bottle, cover top with plastic bag and 6" of No. 24 c-flex micro tubing.
13. Pump micro solution to capacity through tubing leaving no air bubbles. With a small piece of clean No. 24 c-flex attach one end of tubing to another.
14. Place tubing in clean Rubbermaid bag, label and let sit for at least one day.
15. Rinse 6" of No. 24 c-flex micro tubing with DIW before storing.
16. To flush solution out of tubing follow directions 3 - 7.
17. Cover top with plastic bag and 6" of No. 24 c-flex nitric tubing.
18. Pump nitric solution to capacity through tubing leaving no air bubbles.
19. With a small piece of clean No. 24 c-flex nitric attach one end of tubing to another.
20. Place tubing in clean Rubbermaid bag, labeling it and letting it sit for at least 2 days.
21. Rinse 6" of No. 24 c-flex nitric tubing with DIW before storing.
22. Follow steps 3-4 when flushing nitric solution out of tubing.
23. After tubing has been flushed out thoroughly, empty DIW out of tubing and place it in a clean and labeled ice bag.
24. Then close the bag with a cable tie.
25. Store all clean tubing sets in a Rubbermaid bag, in the cleanroom.
26. Be sure to label amount of tubing sets on bag for inventory.

1.3.2 Bottles (125 ml to 1 liter)

1. Fill the bottles to capacity with micro solution, either directly from previous bottle rotation or make a new solution.
2. Shake and let sit in sealed, labeled bags for ***at least one day***.
3. Empty bottles of micro solution in new set of bottles for rotation. To rinse fill bottles 1/4 full with DIW, cap, shake, and empty. ***Do this 3 times***.
4. To transport all bottles in and out of the cleanroom be sure they are in clean and labeled Rubbermaid bags.
5. Next fill the bottles to capacity with 50% nitric solution. It is preferable that this procedure is done in a cleanroom hood. The bottles should be capped tightly. Let bottles sit in nitric solution for ***at least 2 days***.
6. Empty bottles of nitric solution, either into new bottles in rotation or into designated nitric solution bottles.
7. ***Rinse bottles as described in step 3***. If using a cleanroom, be sure to transport all bottles in and out of the cleanroom in clean and labeled Rubbermaid bags.

8. Next fill the bottles to capacity with 10% HCl (hydrochloric) solution. It is preferable that this procedure is performed in a cleanroom hood. The bottles should be capped tightly.
9. Let bottles sit in HCl solution for ***at least two days***.
10. Empty bottles of HCl solution either into new bottles in rotation or designated HCl solution bottles.
11. ***Rinse bottles as described in step 3***. If using a cleanroom, be sure to transport all bottles in and out of the cleanroom in clean and labeled Rubbermaid bags.
12. Dry bottles with the lids on. Let them dry ***at least one day***.
13. Package dry bottles in a Ziplock bag. One gallon size for 1-liter, quart size for 500 ml and 250 ml.
14. Store bottles in a large Rubbermaid bag, labeling amount of bottles on bag for inventory.

1.3.3 Bottles (30 or 60 ml)

1. Place (72, one box) uncapped bottles and lids in clean double ice bag.
2. Fill bag with new micro solution about 1/2 full.
3. Cable tie the bag and label it, make sure there are no air bubbles.
4. Place in a clean Nalgene container and let sit for one day.
5. Empty bottles and lids in a clear Nalgene container.
6. Rinse the bottles and lids ***three times with DIW***.
7. Place bottles and lids in designated container of 50% nitric solution, in the acid hood and let them sit for ***at least 2 days***.
8. After sitting for 2 days remove bottles and lids by placing them in clean Nalgene container. ***Rinse 3 times with DIW***.
9. Place bottles and lids in designated container of 10 % HCl solution, in the acid hood. Let sit for ***at least 2 days***.
10. Remove bottles and lids by placing them in Nalgene container. Also, ***rinse 3 times with DIW***. With the lids on the bottles dry them in the clean room in front of the HEPA filter. Let dry ***at least 1 day***.
11. Package the dry bottles in a labeled Ziplock bag.
12. Store in a large Rubbermaid bag, labeling the amount of bottles on the bag for inventory.

1.3.4 Bottles (2 liters or larger)

1. Wash exterior with Micro solution and designated clean chemistry scrubber.
2. Fill bottles to capacity with micro solution.
3. Shake, label, and let sit in cable tied bags ***at least 1 day***.
4. Empty bottles of micro solution.
5. Rinse bottles 1/4 full with DIW, cap, shake and empty. Do ***this 3 times***. When transporting all bottles be sure they are in clean and labeled Rubbermaid bags.
6. Next fill bottle to capacity with 50 % nitric solution. Make sure procedure is done under vent hood, cap tightly.
7. Let bottles sit in the nitric solution ***at least 2 days***.
8. Empty bottles of nitric solution, either in new bottles in rotation or in designated nitric solution bottles.
9. Rinse bottle as described in step 5. When transporting be sure they are in clean and labeled bags.
10. Next fill bottle to capacity with 10 % HCl solution. Make sure procedure is performed under vent hood, cap tightly.
11. All bottles with HCl solution should be labeled. Let bottles sit in HCl solution ***at least 2 days***.
12. Rinse bottle as described in step 5. When transporting bottles make sure they are in clean and labeled bags.
13. Dry the bottle with lid on.
14. Package dry bottles in labeled ziplock bags.

1.3.5 Cartridge filters

Fill cartridge filters with 10% HCl solution and seal using pre-cleaned No. 24 c-flex tubing. Place in a clean plastic bag and let sit for ***at least 3 days***.

1. Flush cartridge filter with 1 liter of reagent water and re-seal with tubing and let sit ***at least 1 day***.

2. Flush filter with 2 liters of reagent water. Leave filter full.
3. Add 1 ml of ultrapure ammonium hydroxide to filter and re-seal with c-flex tubing. Let filter sit for ***at least 3 days*** before being used for sampling.
4. Filter ***must still be flushed with at least 0.5 to 1 liter*** of the water to be sampled to insure not residual acid is left in the filter prior to the final sample being collected.

1.3.6 Equipment Quality Control

With each cleaning batch of tubing (i.e. typically 10 sets) or bottles (i.e. typically 24-72) a blank is taken. For bottle blanks, reagent water is added to a randomly selected bottle under clean conditions. For tubing sets, reagent water is pumped through the tubing and into a pre-cleaned bottle. The bottle is then treated as a dissolved water sample and analyzed by ICP-MS. If the bottle or sampler blanks show detectable amounts of any element of interest, the batch of affected equipment is re-cleaned.

Section 2 Sampling Methods

The following sections describes grab sampling for metals-in-water and trace elements. The protocols most widely accepted at this time, especially when using the parts-per-billion (µg/L) analytical levels, require the use of "clean" sampling procedures. These sampling procedures help reduce (to the extent possible given current resources) the amount of contamination introduced when collecting water-quality samples in the field. "Clean" sampling procedures involve: (1) using equipment that is constructed of non-contaminating materials and that has been cleaned rigorously before field work and between field sites; (2) handling equipment in a manner that minimizes contamination; (3) collecting, processing, and handling samples in a manner that prevents contamination; and (4) routinely collecting quality-control (QC) samples.

2.1 Grab Sampling

A grab sample is collected in an open container from a single point at, or near, the stream/river/lake/reservoir surface. Grab samples can be collected with a suspended or hand-held polypropylene (Nalgene™) 5-gallon container, disposable bailer, or narrow, open-mouth bottle. If the grab sample is collected by hand-held methods, the sampler should wade where the sample will be collected (preferably at the centroid of flow or mid-channel) and immerse a hand-held narrow-mouth bottle. The sampler should stand downstream of the bottle as it is being filled. Care must be taken to avoid collecting particles that are re-suspended as the result of wading. Various examples of grab samples include: dip, discrete, and pump samples. Dip samples and grab samples and containers (of the appropriate non-contaminating material) such as Nalgene™ buckets or cubitainers are used for grab samples. Discrete or point samples are collected by either (1) lowering a sampler to a specified depth and collecting a sample by first opening, then closing the samplers, or (2) using a single stage sampler, which fills when stream stage rises to a pre-determined height. Thief-type samplers and some pumps are the samplers most often used to collect samples by method 1 above. Although these samplers are designed primarily to sample still waters, they can be adapted to slowly flowing water. Single-stage samplers used in method 2 above include crane-type devices and are useful at stations on flashy streams or other locations where it is difficult to reach a station to manually collect samples. Pump sampling is typically accomplished with suction lift or submersible pump systems designed to collect water-quality samples.

For routine water-quality samples where near-surface water is representative of the water mass, a water sample can be collected by directly immersing the container beneath the water surface to a depth of 1 ft. A bucket can be used to collect a sample if the mixed surface layer is very shallow or accessible only from a bridge. If a bucket is used, caution should be taken to avoid contaminating the sample with debris from the rope and bridge. Care must be taken also to rinse the bucket between stations. In slow-moving rivers, reservoirs, and estuaries, the depth in the mixed layer can be determined from field measurements by locating the thermocline or an abrupt change in specific conductance. In tidally-influenced waterbodies, the mixed surface layer is defined as that portion of the water column from the surface to the depth at which the specific conductance is 6,000 µS/cm greater than the specific conductance at the surface. For mixed surface layer samples (depth >1 ft.), pre-rinse one of the following sample devices at least once with native water before using: submersible pump tube, Kemmerer, or Van Dorn. A minimum volume of 3

L should be collected from each site. Sample containers do not have to be rinsed with site water. Care should be taken at all times during sample collection, handling, and transport to prevent exposure of the sample to direct sunlight. Summary information on sample processing, preservation, and holding times for grab sampling methods, including dissolved metals in water, is contained in Tables 2 and 3.

2.2 Clean Hands/Dirty Hands Techniques for Water-Quality Sampling Overview

"Clean" sampling procedures, including Clean Hands(CH)/Dirty Hands (DH) techniques, are required when collecting samples for metals and other trace elements. CH/DH techniques separate field duties and dedicate one individual as "clean-hands" to tasks related to direct contact with the sample. These techniques are summarized below:

1. CH/DH techniques require two people working together.

2. At the field site, one person is designated as "clean hands" (CH) and the second person as "dirty hands" (DH). Although specific tasks are assigned at the start to CH or DH, some tasks overlap and can be handled by either, as long as contamination is not introduced into samples.

3. Both CH and DH wear appropriate non-contaminating, disposable, powderless gloves during the entire sampling operation and change gloves frequently, usually with each change in task (wearing multiple layers of gloves allows rapid glove changes).

4. **CH takes care of all operations involving equipment that comes into contact with the sample; for example, CH**:

   - ✓ Handles the surface-water sample bottles
   - ✓ Handles the discharge end of the surface-water sample tube or line
   - ✓ Prepares clean work space (either inside vehicle or laboratory prep area)
   - ✓ Sets up processing and preservation chambers
   - ✓ Sets equipment inside chambers (i.e., sample bottles, filtration, and preservation equipment)
   - ✓ Works exclusively inside chambers during collection, processing, and preservation.
   - ✓ Changes chamber covers, as needed
   - ✓ Sets up field-cleaning equipment and cleans equipment

5. **DH takes care of all operations involving contact with potential sources of contamination; for example, DH:**

   - ✓ Works exclusively exterior to processing and preservation chambers
   - ✓ Prepares and operates sampling equipment, including pumps and discrete samplers, peristaltic pump switch, pump controller, and manifold system
   - ✓ Operates cranes, tripods, drill rigs, vehicles, or other support equipment
   - ✓ Handles the compressor or other power supply for samplers
   - ✓ Handles tools such as hammers, wrenches, keys, locks, and sample-flow manifolds
   - ✓ Handles single or multiparameter instruments for field measurements
   - ✓ Handles stream-gaging or water-level equipment
   - ✓ Sets up and calibrates field-measurement instruments
   - ✓ Measures and records water levels and field measurements

2.3 Metals and Trace Elements Sampling Overview

The following field practices are recommended when sampling for metals and trace elements:

1. Think contamination: be aware of and record potential of contamination at each filed site.
2. Wear appropriate non-contaminating, disposable, powderless gloves.
   (a) Change gloves before each step during sample collection (and processing).
   (b) Avoid hand contact with contaminating surfaces (such as equipment, coins and food).
3. Use equipment constructed of materials that are relatively inert with respect to targeted analytes. Metal samplers must be epoxy-coated to prevent trace element contamination.
4. Use only equipment that has been cleaned according to prescribed procedures. Sampling processing equipment should be kept covered (when not dispensing sample).

5. Field rinse equipment, but only as directed. Some equipment for some analytes should not be field rinsed.
6. Use correct sample-handling steps:
   (a) Minimize the number of sample-handling steps
   (b) Use CH/DH techniques as required for parts-per-billion (ppb or µg/L) trace element sampling.
   (c) Adapt CH/DH techniques for all sample types, as required to obtain data of known quality.
   (d) Train for and practice field techniques under supervision before collecting water samples on your own
7. Collect and process samples in a clean enclosure such as a dedicated water-quality field vehicle or field processing chamber. Metallic objects, dirt, oil residue, engine exhaust, and food can all be sources of contamination. Sample processing chambers can be fashioned from a polyvinyl chloride frame and clear plastic bag.
8. Filter samples for *dissolved trace elements* and *metals* as soon as practical after collection with disposable, tortuous path, capsule filter (effective size of 0.45 µm; 15 mm diameter or larger, Gelman Supor™ 12175, or equivalent [Ref. EPA Method 1669]). A variable speed, battery-operated pump fitted with a peristaltic pump head that forces the sample through Tygon™ or Teflon™ tubing is recommended. Filtered samples should be preserved with (1+1), ~7.7N Ultrex™, Ultrapure Reagent nitric acid (HNO_3), to a pH ≤ 2.0. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample. For field purposes, the nitric acid can be stored on quantities of 2 mL in polypropylene vials.
9. Collect a sufficient number of appropriate types of QC samples. QC samples should be reviewed to determine if cleaning procedures are sufficient and contamination has been minimized.

Specific details regarding grab sample collection methods, preservation, storage, and handling requirements are summarized in Tables 2 and 3.

2.4 Dissolved Metals-in-Water Collection Technique

Collecting metals-in-water requires two people. One person, designated as "Clean Hands," is the only one in direct contact with the sampling container, tubing, and filter (or anything that touches the ambient or blank water). The second person, or "Dirty Hands." sets up the apparatus and operates the pump. Both CH and DH wear powder-free latex gloves during the sample collection process.

Sample Collection

At the site, DH sets up the pump while the CH takes a bottle from the plastic bag and places it in a "container holder." A container holder can be anything nonmetal that supports the bottle, freeing up the collector's hands. CH takes the end of the tubing with the filter attached out of the bag and places it in the pump head. The outlet end is approximately 18 inches from the pump. The other end is long enough to easily reach beneath the surface water. DH closes the pump head, locking the tubing in place.

CH takes the other end if the tubing, removes the plastic cover from the end of the tubing and places it in a "tubing holder." The tubing holder can be a PVC pipe, or something similar (nonmetallic), can be used to hold and extend the tubing beneath the water surface.

DH immerses the intake tube directly into the water and operates the pump to flush the tube and filter with the filter held upright. CH removes the cap from the sample container to fill with filtrate leaving some head space. CH puts the cap back on the container and places it back in the plastic bag. Whenever CH touches the boat or equipment, which may be contaminated, gloves should be changed immediately.

Sample Preservation

If not using a commercially purchased pre-acidified container, filtered metals-in-water samples should be preserved with (1+1), ~7.7N Ultrex™, Ultrapure Reagent nitric acid (HNO_3), to a pH ≤ 2.0. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample. For field purposes, the nitric acid can be stored in quantities of 2 mL in polypropylene vials. Holding times for acid preserved samples is six months except for mercury which is 28 days. After collecting the sample and adding the preservative, the container is placed back in a plastic bag for shipping. This is to prevent possible contamination from other samples in the ice chest.

Trivalent Chromium due to the complexity of the preservation method, it is advised that the sample we shipped to the laboratory unpreserved.

Hexavalent Chromium acidification alters the hexavalent form of chromium, a separate (unacidified) sample must be submitted if hexavalent chromium is to be analyzed. Filter, add 1 mL NaOH per 125 mL of sample. Submit a minimum of 500 mL sample. The sample is collected, placed on ice, and shipped to the laboratory in time for analysis to begin within 24-hours of collection. The laboratory should be notified when a hexavalent chromium sample will arrive. Hexavalent chromium is not usually analyzed on unfiltered samples.

Sample Container Label

Label each container with the tag number and the type of sample. Since the sample has been filtered and preserved, this information should be noted on the laboratory analyses request form.

Field Blanks and Replicates

Field blanks should be prepared immediately before collecting and processing an environmental sample at a selected site. All equipment should have been cleaned, either in the field or preferably in the laboratory following guidelines outlined in this guidance. The water used for field blanks should be metals free. Equipment need not be cleaned nor filters changed between processing the field blank and the environmental sample. However, certain pieces of equipment should be cleaned or changed if they have obviously been contaminated or if there is a significant potential for contamination. The sequential replicates are collected as you would collect a normal sample.

The sample collection method outlined for dissolved metals-in-water samples is followed for the field blank with the following exceptions:

CH opens a container of blank water (metals-free DI water). DH removes the plastic cover from the end of the tubing and inserts the tubing into the blank water container. DH holds the tubing in place.

CH takes the plastic cover off the other end of the tubing. DH turns on the pump and flushes a small amount of water through the filter to purge it for dissolved metals.

CH removes the cap from the sample container and uses the pump to fill with metals-free DI water. CH puts the cap back on the container and places it in the plastic bag.

2.5 Companion Samples for Metals-in-Water

Request a hardness analyses whenever metals-in-water are to be analyzed from an inland site (estuaries sites do not require hardness analysis). Typically, the hardness can be calculated from the analysis of calcium and magnesium. Sample holding time for unpreserved samples is 2 days under refrigeration. Hardness samples can be preserved giving longer holding times, but they must be filtered before the acid preservative is added. Filter, then preserve with 2 mL of concentrated H_2SO_4 or 5 mL of concentrated HNO_3 per liter of sample.

Section 3 Types of Quality-Control Collection Requirements for Metals-in-Water

Three types of QC samples are routinely collected for metals-in-water studies. Blanks and spikes are used to estimate bias. Replicates are used to estimate variability.

A **blank** is a water sample that is intended to be free of the analytes of interest. Blank samples are analyzed to test for bias that could result from contamination of environmental samples by the analytes of interest during any stage of sample collection, processing, and analysis. A field blank is prepared in the field and used to demonstrate that: (1) Equipment has been adequately cleaned to remove contamination introduced by samples obtained at previous sites; (2) sample collection and processing have not resulted in contamination; and (3) sample handling and transport have not introduced contamination. In addition, because the field blank is treated like an environmental sample at the laboratory, it includes potential contamination introduced during laboratory handling and analysis.

A **field blank** is used to demonstrate that the sample-collection and sample-processing equipment are not introducing contamination. Field blanks can be prepared using individual pieces of the collection and processing equipment. For example, a sample prepared by exposing the blank solution just to the filter apparatus would be a filter blank. In metals-in-water, the only type of field blank that is routinely prepared is by filtering a quantity of metals free water through the tubing and filter apparatus. This is used to determine whether the tubing and filter are introducing contamination into the sample.

A **trip blank** is a sample of analyte-free water that is prepared in the laboratory or in the office. It is transported, unopened, to the field with other sample containers and is shipped to the laboratory for analysis with the collected samples. Trip blanks are used to identify contamination that might occur during

sample transport and analysis rather than as a result of sample collection and processing in the field. Because the primary source of this contamination is airborne, trip blanks are normally prepared for VOCs.

Replicates are two or more samples collected or processed so that the samples are considered to be essentially identical in composition. **Split replicates** are prepared by dividing a single volume of water into multiple samples. These replicates provide a measure of variability introduced during sample processing and analysis. **Concurrent replicates** are multiple samples collected from an environmental matrix as closely as possible to the same location and time. These replicates account for the variability measured by split replicates and the additional variability introduced by sample collection. Depending on sampling procedures, concurrent replicates also might include an unknown amount of short-term environmental variability. **Sequential replicates** are multiple samples collected at the same location, but at slightly different times, generally one right after the other. These replicates provide a measure of the same sources of variability as concurrent replicates and the additional variability associated with short-term environmental fluctuation.

For purposes of this SOP, the term "replicate" is used to refer to all similarly collected or processed samples. The term "primary environmental sample" and "duplicate environmental sample" are used to identify particular samples in a replicate pair.

3.1 Field Blanks

Field blanks should be prepared immediately before collecting and processing an environmental sample at a selected site. All equipment should have been cleaned, either in the field or preferably in the laboratory following guidelines outlined in this guidance. The water used for field blanks should be metals free. Equipment need not be cleaned nor filters changed between processing the field blank and the environmental sample. However, certain pieces of equipment should be cleaned or changed if they have obviously been contaminated or if there is a significant potential for contamination.

3.2 Replicates

Replicates for analysis of metals-in-water are produced by splitting a single, large volume of water, collected from the waterbody, into two samples (one primary and one duplicate). The split replicates will allow assessment of sources of variability (sample processing, handling and analysis) that can be controlled by field and laboratory procedures. Sequential replicates are collected for the purpose of estimating the magnitude of variation of the sample.

Replicates should be targeted at sites and times where concentrations of at least some target analytes are expected to exceed detection limits. If concentrations of all target analytes are expected to be less than detection, collection of replicates should be deferred until conditions are more favorable for detection or canceled since non-detect information will not provide the requisite information to estimate the magnitude of variation of the sample. Attempt to collect replicates over the range of detectable concentrations expected within the study area, but give greater emphasis to collecting replicates at high concentrations. Replicate samples need not be collected uniformly throughout the sampling period or at all surface water sites; however, they should be scheduled to cover a wide range of hydrologic conditions.

3.3 Quality Control (QC) Data Assessment Guidelines

Analyses of field/equipment blanks associated with the equipment preparation and field sampling are required to demonstrate freedom from contamination introduced during the equipment cleaning and actual sampling. The following sections describe the required types, procedures, and criteria for analyzing field and equipment blanks.

3.3.1 Analyze the field blank(s)(samples collected from the same site at the same time) shipped with each set of samples. Analyze the blank immediately before analyzing the samples in the batch.

3.3.2 If the metal of interest or any potentially interfering substance is found in the field/equipment blank at a concentration equal to or greater than the method limit (ML), or

greater than one-fifth the level in the associated sample, whichever is greater, results for associated samples may be the result of contamination and may not be reported for regulatory compliance purposes.

3.3.3 Alternatively, if a sufficient number of field/equipment blanks (three minimum) are analyzed to characterize the nature of the field/equipment blank, the average concentration plus two standard deviations must be less than the regulatory compliance level or less than one-half the level in the associated sample, whichever is greater.

3.3.4 If contamination of the field blanks and associated samples is known or suspected, the laboratory should communicate this to the sampling team so that the source of contamination can be identified and corrective measures taken before the next sampling event.

3.3.5 Both the ***field*** and ***equipment blanks*** are defined in Section 3.0

3.3.6 Data Use and Acceptability

Regardless of the field sampling method(s) used, all data will be deemed acceptable and used for Section 303(d) and 305(b) decisions unless the data has been deemed unacceptable due to the stated QC guidelines, Sections 3.3.2 and 3.3.3. If the data does not meet the QC guidelines, it will be censored (disregarded) for use in Section 303(d) and 305(b) decisions.

3.3.7 Toxicity Assessment Guidelines

Those samples that are deemed acceptable based on the stated QC guidelines will be assessed as follows in making designated use attainment decisions:

Fully Supporting - For any one pollutant, no more than 1 exceedance of acute criteria (State's criteria maximum concentration) within a 3-year period and no more than 1 exceedance of chronic criteria (State's criteria continuous concentration) within a 3-year period.

Table 1. Collection frequencies for routine quality-control samples
[- -, denotes no samples required]

Constituent or group	Number of quality-control samples per total number of environmental samples (at all surface-water sites, each year)			
	Field blanks	Trip blanks	Replicate field matrix spikes	Replicates
Dis. Metals-in-water	1 in 10 samples②	- -	- -	1 in 10① samples
Trace elements	1 in 10 samples	- -	- -	1 in 10① samples

①A minimum of three **sequential replicates** will be collected. However, if, in the judgement of the project manager, the concentrations of all target analytes are expected to be less than detection, collection of replicates will be deferred until conditions are more favorable for detection or canceled since non-detect information will not provide the requisite information to estimate the magnitude of variation of the sample.
②If the metal of interest or any potentially interfering substance is found in the field/equipment blank at a concentration equal to or greater than the method limit (ML), or greater than one-fifth the level in the associated sample, whichever is greater, results for associated samples may be the result of contamination and may not be reported for regulatory compliance purposes. Alternatively, if a sufficient number of field/equipment blanks (three minimum) are analyzed to characterize the nature of the field/equipment blank, the average concentration plus two standard deviations must be less than the regulatory compliance level or less than one-half the level in the associated sample, whichever is greater.

Table 2. General Guidelines for selecting equipment on the basis of construction material and target analyte(s)

[✔, generally appropriate for use shown; Si, silica; Cr, chromium; Ni, nickel; Fe, iron; Mn, manganese; Mo, molybdenum; CFC, chlorofluorocarbon; B, boron]

Construction material for sampling equipment		Target analyte(s)	
Material	**Description**	**Inorganic**	**Organic**
	Plastics[1]		
Fluorocarbon ploymers[2] (other varies available for differing applications)	Chemically inert for most analytes	✔ (potential source of fluoride)	✔ (Sorption of some organics)
Polypropylene	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polypropylene (linear)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polyvinyl chloride (PVC)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Silicone	Very porous. Relatively inert for most inorganic analytes	✔ (potential source of Si)	**Do not use**
	Metals		
Stainless steel 316 (SS 316)	SS-316-metal having the greatest corrosion resistance. Comes in various grades. Used for submersible pump casing.	✔ (Potential source of Cr, Ni, Fe, and possible Mn and Mo) **Do not use** for surface water unless encasted in plastic.	✔ Do not use if corroded[3]
Stainless steel 304	Similar to SS-316, but less corrosion resistant	**Do not use**	✔ Do not use if corroded[3]
Other metals: brass, iron, copper, aluminum, galvanized and carbon steels	Refrigeration-grade copper or aluminum tubing are used routinely for collection of CFC samples	**Do not use**	✔ Routinely used for CFCs Do not use if corroded[3]
	Glass		
Glass, borosilicate (laboratory grade)	Relatively inert. Potential sorption of analytes	✔ **Do not use** for trace element analyses. Potential source of B and Si	✔

[1]Plastic used in connection with inorganic trace-element sampling should be uncolored or white. Tubing used for trace metal sampling should be cleaned by soaking in 5-10 percent HCl solution for 8-24 hours, rinsing with reagent water (metals free) and allowed to air dry in mercury-free environment. After drying, the tubing is doubled-bagged in clear polyethylene bags, serialized with a unique number, and stored until used.

[2] Fluorocarbon polymers include materials such as Teflon™, Kynar™, and Tefzel™ that are relatively inert for sampling inorganic or organic analytes. Only fluoropolymer should be used for samples that will analyzed for mercury because mercury vapors can diffuse in or out of other materials, resulting in either contaminated or biased results.

[3] Corroded/weathered surfaces are active sorption sites for organic compounds.

Table 3. Summary of grab sample collection methods, preservation, storage and handling requirements

PARAMETERS	CONTAINERS	SAMPLE VOLUME (mL)	PRESERVATION	MAXIMUM HOLDING TIME
		WATER		
		ROUTINE WATER SAMPLE		
Alkalinity	Cubitainer or Glass	100	Cool to 4 ^{0}C, dark	14 days
Total Suspended Solids/Suspended Solids	Cubitainer or Glass	400	Cool to 4 ^{0}C, dark	7 days
Chloride (Cl)	Cubitainer or Glass	100	None required	28 days
Sulfate (SO_4)	Cubitainer or Glass	100	Cool to 4 ^{0}C, dark	28 days
Orthophosphate (OPO_4)	Cubitainer or Glass	150	Filter ASAP; Cool to 4 ^{0}C, dark	48 hours
Nitrate + Nitrite ($NO_3 + NO_2$)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 ^{0}C, dark	28 days
Ammonia (NH_3)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 ^{0}C, dark	28 days
Total Phosphorus (TPO_4)	Cubitainer or Glass	150	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 ^{0}C, dark	28 days
Total Organic Carbon (TOC)	Cubitainer or Glass	100	1-2 mL conc. H_2SO_4 to pH <2, and Cool to 4 ^{0}C, dark	28 days
Chlorophyll *a*	Quart cubitainer	1,000	Cool to 4 ^{0}C, dark	Filter 48 hours Filters may be stored frozen up to 30 days
Nitrite	Quart cubitainer	50	Cool to 4 ^{0}C, dark	48 hours
Total Dissolved Solids	Quart cubitainer	250	Cool to 4 ^{0}C, dark	7 days
Hardness	Quart cubitainer	250	2 mL conc. HNO_3 to pH<2; Cool to 4 ^{0}C, dark **OR** 2 mL conc. H_2SO_4 to pH <2; Cool to 4 ^{0}C, dark	6 months

ROUTINE WATER SAMPLE COLLECTION PROCEDURE

- Label container before collection with a unique sample identifier number, Station Location, Date and Sample Type
- Place an **X** on the container lid to identify the acidified sample.
- Open containers by pulling apart. Pre-rinsing cubitainers with ambient water is not necessary.
- Fill each container with ambient water by submerging container approximately one foot below the surface mid-stream until filled.
- Place sample on ice immediately. Acidify the **X** container as soon as possible.
- Place on ice and ship as soon as possible.

Table 3. Summary of grab sample collection methods, preservation, storage and handling requirements–Continued

PARAMETERS	CONTAINERS	SAMPLE VOLUME (mL)	PRESERVATION	MAXIMUM HOLDING TIME
		WATER		

NON-ROUTINE WATER SAMPLES				
OIL AND GREASE	Glass container with teflon lined lid rinsed with hexane or methylene chloride	1,000	2 mL conc. H_2SO_4 to pH <2; cool to 4 ^{0}C, dark	28 days
PHENOLS	Glass container with teflon lined lid	1,000	2 mL conc. H_2SO_4 to pH <2; cool to 4 ^{0}C, dark	28 days
BIOCHEMICAL OXYGEN DEMAND	Gallon cubitainer	> 4,000	Cool to 4 ^{0}C; add 1g FAS crystals per liter if residual chlorine present	48 hours
CHEMICAL OXYGEN DEMAND	Quart cubitainer	110	2 mL conc. H_2SO_4 to pH <2; cool to 4 ^{0}C, dark	28 days
METALS-IN-WATER				
DISSOLVED (except Hg)	HNO_3 cleaned quart plastic container	1,000	Filter at sample site with 0.45 micron in-line filter[1] into ultra-pure[2] HNO_3 preacidified container to pH<2	6 months
DISSOLVED MERCURY	HNO_3 cleaned quart plastic container	1,000	Filter at sample site with 0.45 micron in-line filter[1] into ultra-pure[2] HNO_3 preacidified container to pH<2	28 days
TOTAL (except Hg)	HNO_3 cleaned quart plastic container	1,000	Preacidified container with 5 mL ultra-pure[2] HNO_3 to pH<2	6 months
TOTAL MERCURY (Hg)	HNO_3 cleaned quart plastic container	600	Preacidified container with 5 mL ultra-pure[2] HNO_3 to pH<2	28 days
HEXAVALENT CHROMIUM (filtered)	Plastic or glass	600	Cool to 4 ^{0}C, dark	24 hours; must notify lab in advance

METALS-IN-WATER SAMPLE COLLECTION PROCEDURES

DISSOLVED METALS (includes Hexavalent Chromium)
- Put on ***powder-free*** latex, polyethylene, or vinyl gloves using Clean Hands/Dirty Hands technique.
- Assemble pump[3], tubing, and filter.
- Immerse intake tubing directly into water 1ft. and pump approx. 500 mL of ambient water to flush tubing and filter.
- Fill precleaned, preacidified container with 600-1,000 mL of filtrate leaving some head space.

TOTAL METALS
- Put on ***powder-free*** latex, polyethylene, or vinyl gloves using Clean Hands/Dirty Hands technique.
- Assemble pump, and tubing without filter.
- Immerse intake tubing directly into water 1ft. and pump approx. 500 mL of ambient water to flush tubing
- Fill precleaned, preacidified container with 600-1,000 mL of filtrate leaving some head space.

NOTES

[1]Capsule Filter: 15 mm diameter or larger, tortuous path capsule filters, Gelman Supor™ 12175, or equivalent (Ref. EPA Method 1669).

[2]Nitric Acid, Ultra-pure, commercially known as Ultrex™, Ultrapure Reagent.

[3]Pump and pump apparatus–Required for use with the container method. Peristaltic pump–115 a.c., 12 volt d.c., internal battery, variable speed, single head, Cole-Parmer, portable, Masterflex L/S™, Catalog No. H-07570-10 drive with Quick Load pump head, Cat. No. H-07021-24, or equivalent (Ref. EPA Method 1669).

Table 3. Summary of grab sample collection methods, preservation, storage and handling requirements–Continued

PARAMETERS	CONTAINERS	SAMPLE VOLUME (mL)	PRESERVATION	MAXIMUM HOLDING TIME
ORGANICS/PESTICIDES-IN-WATER				
VOLATILE ORGANICS (VOA)	Two 40-mL VOA vials	80	Cool to 4 ^{0}C, dark; or 2-4 drops[1] HCl to pH<2, cool to 4 ^{0}C, dark for BTEX	14 days
ORGANICS		1,000	Cool to 4 ^{0}C, dark	7 days until extraction and 40 days after extraction
PESTICIDES & HEBICIDES Organophosphorus Pesticides Organochlorine Pesticcides Chlorinated Herbicides	1-qt. glass container with teflon lined lid per sample type; *must be prerinsed with hexane*, acetone, or methylene *chloride*	**Each sample type requires 1,000 mL in a separate container**	If chlorine is present, add 0.1 g sodium thiosulfate	
SEMI-VOLATILE ORGANICS				
ORGANICS-IN-WATER COLLECTION PROCEDURES				
2Label each container before collection with tag no./unique sample identifier number, Station Location, Date, and "ORGANICS: Organophosphorus Pesticides, Organochlorine Pesticides, or Chlorinated Herbicides" or "SEMI-VOLATILE" (depending on the sample type). ◦Fill to the top. Put in dark and on ice. ◦Fill quart container(s) to the top. Put in dark and on ice.				
BIOLOGICAL				
TOXICITY IN WATER	Two 1-gallon cubitainers	8,000 mL	Cool to 4 ^{0}C, dark	36 hours
TOXICITY SAMPLE COLLECTION PROCEDURES				
WATER ◦Label containers before collection with Station Location, Date, and Sample Type. ◦Open cubitainer by pulling apart. Pre-rinsing cubitainers with ambient water is not necessary. ◦Fill each container with ambient water by submerging container approx. 1-ft. below the surface mid-stream until filled. ◦Place on ice and ship as soon as possible.				
NOTES or d preserving with HCl, discuss with laboratory personnel; preserved samples may cause damage to analytical equipment. If sample is analyzed within 48 hours, preservation may not be required.				

Designation: E 1676 – 04

Standard Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia Fetida* and the Enchytraeid Potworm *Enchytraeus albidus*[1]

This standard is issued under the fixed designation E 1676; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers procedures for obtaining laboratory data to evaluate the adverse effects of contaminants (for example, chemicals or biomolecules) associated with soil to earthworms (Family Lumbricidae) and potworms (Family Enchytraeidae) from soil toxicity or bioaccumulation tests. The methods are designed to assess lethal or sublethal toxic effects on earthworms or bioaccumulation of contaminants in short-term tests (7 to 28 days) or on potworms in short to long-term tests (14 to 42 days) in terrestrial systems. Soils to be tested may be *(1)* reference soils or potentially toxic site soils; *(2)* artificial, reference, or site soils spiked with compounds; *(3)* site soils diluted with reference soils; or *(4)* site or reference soils diluted with artificial soil. Test procedures are described for the species *Eisenia fetida* (see Annex A1) and for the species *Enchytraeus albidus* (see Annex A4). Methods described in this guide may also be useful for conducting soil toxicity tests with other lumbricid and enchytraeid terrestrial species, although modifications may be necessary.

1.2 Modification of these procedures might be justified by special needs. The results of tests conducted using atypical procedures may not be comparable to results using this guide. Comparison of results obtained using modified and unmodified versions of these procedures might provide useful information concerning new concepts and procedures for conducting soil toxicity and bioaccumulation tests with terrestrial worms.

1.3 The results from field-collected soils used in toxicity tests to determine a spatial or temporal distribution of soil toxicity may be reported in terms of the biological effects on survival or sublethal endpoints (see Section 14). These procedures can be used with appropriate modifications to conduct soil toxicity tests when factors such as temperature, pH, and soil characteristics (for example, particle size, organic matter content, and clay content) are of interest or when there is a need to test such materials as sewage sludge and oils. These methods might also be useful for conducting bioaccumulation tests.

1.4 The results of toxicity tests with *(1)* materials (for example, chemicals or waste mixtures) added experimentally to artificial soil, reference soils, or site soils, *(2)* site soils diluted with reference soils, and *(3)* site or reference soils diluted with artificial soil, so as to create a series of concentrations, may be reported in terms of an LC50 (median lethal concentration) and sometimes an EC50 (median effect concentration). Test results may be reported in terms of NOEC (no observed effect concentration), LOEC (lowest observed effect concentration) or as an ECx (concentration where x % reduction of a biological effect occurs. Bioaccumulation test results are reported as the magnitude of contaminant concentration above either the Day 0 tissue baseline analysis or the Day 28 tissues from the negative control or reference soil (that is, 2×, 5×, 10×) (see A3.9).

1.5 This guide is arranged as follows:

[1] This guide is under the jurisdiction of ASTM Committee E47 on Biological Effects and Environmental Fate and is the direct responsibility of Subcommittee E47.02 on Terrestrial Assessment and Toxicology.

An ASTM guide is defined as a series of options or instructions that do not recommend a specific course of action.

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1.6 The values stated in SI units are to be regarded as the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* While some safety considerations are included in this guide, it is beyond the scope of this standard to encompass all safety requirements necessary to conduct soil toxicity tests. Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluid

D 4447 Guide for the Disposal of Laboratory Chemicals and Samples

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)

E 943 Terminology Relating to Biological Effects and Environmental Fate

E 1023 Guide for Assessing the Hazard of a Material to Aquatic Organisms and Their Uses

E 1383 Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates

E 1688 Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates

E 1706 Test Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Fresh Water Invertebrates

3. Terminology

3.1 *Definitions:*

3.1.1 The words "must," "should," "may,"" can," and "might" have very specific meanings in this guide. "Must" is used to express an absolute requirement, that is, to state that the test must be designed to satisfy the specified condition, unless the purpose of the test requires a different design. "Must" is used only in connection with the factors that relate directly to the acceptability of the test (see Section 13). "Should" is used to state that the specified condition is recommended and ought to be met if possible. Although a violation of one "should" is rarely a serious matter, the violation of several will often render the results questionable. Terms such as "is desirable," "is often desirable," and "might be desirable" are used in connection with less important factors. "May" is used to mean "is (are) allowed to," "can" is used to mean "is (are) able to," and "might" is used to mean "could possibly." Thus, the classic distinction between "may" and "can" is preserved, and "might" is never used as a synonym for either "may" or "can."

3.1.2 For definitions of terms used in this guide, refer to Terminology E 943 and Guide E 1023. For an explanation of units and symbols, refer to Practice E 380.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *artificial soil*—a synthetic soil, prepared with a specific formulation, designed to simulate a natural soil (see Annex A2). Artificial soil may be used as a diluent medium to prepare concentrations of site or reference soil and may be used as a negative control medium.

3.2.2 *batch*—the total amount of test soil prepared for each concentration in a test. A batch is any hydrated test soil ready for separation into replicates.

3.2.3 *bioaccumulation*—the net accumulation of a substance by an organism as a result of uptake from all environmental sources. (See Guide E 1688.)

3.2.4 *bioaccumulation factor (BAF)*—the ratio of tissue residue to sediment contaminant concentration at steady-state. (See Guide E 1688.)

3.2.5 *bioaccumulation potential*—a qualitative assessment of whether a contaminant in a particular sediment is bioavailable. (See Guide E 1688.)

3.2.6 *bioconcentration*—the net assimilation of a substance by an organism as a result of uptake directly from aqueous solution. (See Guide E 1688.)

3.2.7 *bioconcentration factor (BCF)*—the ratio of tissue residue to water contaminant concentration as steady-state. (See Guide E 1688.)

3.2.8 *biota-sediment accumulation factor (BSAF)*—the ratio of lipid-normalized tissue residue to organic carbon-normalized sediment contaminant concentration at steady state, with units of g-carbon/g-lipid. (See Guide E 1688.)

3.2.9 *clitellum*—the fleshy "ring" or "saddle" of glandular tissue found on certain mid-body segments of oligochaete (Lumbricidae and Enchytraeidae) worms. It is the most visible feature of an adult earthworm or potworm and secretes the cocoon into which eggs and sperm are deposited.

3.2.10 *concentration*—the ratio of the weight of test materials to the weight of soil (artificial, reference, or site), usually expressed on a dry weight basis as percent or milligram/kilogram.

3.2.11 *depuration*—loss of a substance from an organism as a result of any active (for example, metabolic breakdown) or passive process.

3.2.12 *diluent soil*—the artificial or reference soil used to dilute site soils.

3.2.13 *enchytraeid*—potworm members of the Family Enchytraeidae of the Class Oligochaeta of the Phylum Annelida.

3.2.14 *hydration water*—water used to hydrate test soils to create an environment with a moisture level suitable for the species being tested. The water used for hydration is often test water (see 3.2.27); however, depending on the nature of the test being implemented, site surface water or groundwater may also be utilized for hydration.

3.2.15 *lumbricid*—earthworm members of the Family Lubricidae of the Class Oligochaeta of the Phylum Annelida.

3.2.16 *negative control soil*—artificial or reference soil to be used for evaluating the acceptability of a test.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.17 *reference soil*—a field-collected soil that has physicochemical and biological properties as similar as possible to the site soil but does not contain the potentially toxic compounds of the site soil. It is used to describe matrix effects on the test in question. It may be used as a diluent medium to prepare concentrations of site soil and may be used as a negative control medium.

3.2.18 *sampling station*—a specific location, within a site or sampling unit, depending on the field study design, at which soil is collected for chemical, physical, and biological evaluation.

3.2.19 *sampling unit*—an area of land within a site distinguished by habitat and topography.

3.2.20 *site*—a delineated tract of land that is being considered as a study area, usually from the standpoint of its being potentially affected by xenobiotics.

3.2.21 *site soil*—a soil collected from the field to be evaluated for potential toxicity. A site soil may be a naturally occurring soil or one that has been influenced by xenobiotics.

3.2.22 *soil*—sediments or other unconsolidated accumulations of solid particles produced by the physical and chemical disintegration of rocks, and that may or may not contain organic material. (See Terminology D 653.)

3.2.23 *spiking*—the experimental addition of a test material to an artificial, site, or reference soil, such that the toxicity of the material added can be determined. After the test material is added, which may involve a solvent carrier, the soil is mixed thoroughly to distribute the test material evenly throughout the soil.

3.2.24 *test chamber*—an enclosed space or compartment in which environmental parameters such as temperature and lighting are controlled (for example, incubator or modified room). Test containers are placed in the test chamber for biological evaluation.

3.2.25 *test container*—the experimental unit; the smallest physical entity to which treatments can be assigned independently.

3.2.26 *test soil*—a soil prepared to receive a test organism. Site or reference soil mixed with artificial soil or reference soil mixed with site soil in known concentrations for evaluation are test soils. Artificial, site, or reference soils spiked with test materials such as chemicals, oils, or manufacturing products are test soils. Once a site, reference, or artificial soil is hydrated, even though it is not mixed with artificial or reference soil or spiked with a material, it may be called a test soil.

3.2.27 *test water*—water used to prepare stock solutions, rinse test organisms, rinse glassware, and apparatus or for any other purpose associated with the test procedures or culture of the test organism. Test water must be deionized or distilled water or better, such as reagent-grade water produced by a system of reverse osmosis, carbon, and ion-exchange cartridges.

4. Summary of Guide

4.1 The toxicity of test soils or the bioavailability of contaminants are assessed during the continuous exposure of terrestrial organisms. Soils tested may be the following: (*1*) soils collected from potentially contaminated sites, (*2*) soils collected from reference sites, (*3*) artificial soil (see Annex A2) spiked with compounds, (*4*) site soil spiked with compounds, (*5*) reference soil spiked with compounds, (*6*) site soil diluted with artificial soil, (*7*) site soil diluted with reference soil, or (*8*) reference soil diluted with artificial soil. A negative control of artificial or reference soil is used for the following: (*1*) to yield a measure of the acceptability of the test; (*2*) to provide evidence of the health and relative quality of the test organisms; (*3*) to determine the suitability of test conditions, food, and handling procedures; and (*4*) to provide a basis for interpreting data obtained from the test soils. Specified data are obtained to determine the toxic effects on survival or sublethal endpoints for 7 to 28-day exposures or containment bioaccumulation for 28-day exposures to terrestrial lumbricids and the toxic effects on survival or sublethal endpoints for 4 to 42-day exposures to enchytraeids.

4.2 *Summary of Changes*—This current version of the standard is a revision of the E 1676-97 version. Changes made since 1997 involve toxicity testing procedures for the Enchytraied potworm, *Enchytraeus albidus*. There has been an additional annex added (Annex A4) and the main document has been modified to include this species.

5. Significance and Use

5.1 Soil toxicity tests provide information concerning the toxicity and bioavailability of chemicals associated with soils to terrestrial organisms. As important members of the soil fauna, lumbricid earthworms and enchytraeid potworms have a number of characteristics that make them appropriate organisms for use in the assessment of potentially hazardous soils. Earthworms may ingest large quantities of soil, have a close relationship with other soil biomasses (for example, invertebrates, roots, humus, litter, and microorganisms), constitute up to 92 % of the invertebrate biomass of soil, and are important in recycling nutrients **(1, 2)**.[3] Enchytraeids contribute up to 5.2 % of soil respiration, constitute the second-highest biomass in many soils (the highest in acid soils in which earthworms are lacking) and effect considerably nutrient cycling and community metabolism **(94-96)**. Earthworms and potworms accumulate and are affected by a variety of organic and inorganic compounds **(2-7, 97-100)**. In addition, earthworms and potworms are important in terrestrial food webs, constituting a food source for a very wide variety of organisms, including birds, mammals, reptiles, amphibians, fish, insects, nematodes, and centipedes **(8, 9, 94)**. A major change in the abundance of soil invertebrates such as lumbricids or enchytraeids, either as a food source or as organisms functioning properly in trophic energy transfer and nutrient cycling, could have serious adverse ecological effects on the entire terrestrial system.

5.2 A number of species of lumbricids and enchytraeid worms have been used in field and laboratory investigations in the United States and Europe. Although the sensitivity of various lumbricid species to specific chemicals may vary, from their study of four species of earthworms (including *E. fetida*) exposed to ten organic compounds representing six classes of

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

chemicals, Neuhauser, et al **(4)** suggest that the selection of earthworm test species does not affect the assessment of a chemical's toxicity markedly. The sensitivity of various enchytraeid species has not been investigated in a comparable way so far, but ecological importance and practicability reasons favor strongly the selection of a species belonging to the genus *Enchytraeus*.

5.2.1 *E. fetida* is a species whose natural habitats are those of very high organic matter such as composts and manure piles. It was selected as the test species because it (*1*) is bred in the laboratory easily; (*2*) is the earthworm species used most commonly in laboratory experiments **(10)**; (*3*) has been studied extensively, producing a data pool on the toxicity and bioaccumulation of a variety of compounds **(2, 4, 5, 11-16)**; (*4*) has been approved for use in toxicity testing by the European Union (EU) and the Organization for Economic Cooperation and Development (OECD); and (*5*) has been used by the Environmental Protection Agency (EPA) for the toxicity screening of hazardous waste sites **(17)**.

5.2.2 The recommended enchytraeid test species is *Enchytraeus albidus* Henle 1837 (white potworm). *E. albidus* is one of the biggest (up to 15 mm) species of the oligochaete family Enchytraeidae and it is distributed world-wide **(101, 102)**. *E. albidus* is found in marine, limnic, and terrestrial habitats, mainly in decaying organic matter (seaweed, compost) and rarely in meadows **(95, 102)**. This broad ecological tolerance and some morphological variations might indicate that there are different races for this species. *E. albidus* is commercially available, sold as food for fish, can be bred easily in a wide range of organic waste materials and has a short life cycle (33 to 74 days; **103, 104**). *E. albidus* was studied in various tests, which covered a wide range of compounds **(104-106)**. In addition, it is currently under investigation for use in toxicity testing and soil quality assessment by the European Union (EU), the Organization for Economic Cooperation and Development (OECD), and the International Organization for Standardization (ISO). Other species of the genus *Enchytraeus* are also suitable, for example, *E. buchholzi* Vejdovsky 1879 or *E. crypticus* Westheide and Graefe 1992 (see Annex A4). Those species are true soil inhabitants and are smaller in size. Other species of *Enchytraeus* may be used, but they should be identified clearly and the rationale for their selection should be reported.

5.3 Results from soil toxicity tests might be an important consideration when assessing the hazards of materials to terrestrial organisms.

5.4 Information might also be obtained on the bioaccumulation of chemicals associated with soil by analysis of animal tissues for the chemicals being monitored. These results are useful for studying the biological availability of chemicals.

5.5 The soil toxicity test might be used to determine the temporal or spatial distribution of soil toxicity. Test methods can be used to detect horizontal and vertical gradients in toxicity.

5.6 Results of soil toxicity tests could be used to compare the sensitivities of different species.

5.7 An understanding of the effect of these parameters on toxicity and bioaccumulation may be gained by varying soil characteristics such as pH, clay content, and organic material.

5.8 Results of soil toxicity tests may be useful in helping to predict the effects likely to occur with terrestrial organisms in field situations.

5.8.1 Field surveys can be designed to provide either a qualitative or quantitative evaluation of biological effects within a site or among sites.

5.8.2 Soil surveys evaluating biological effects are usually part of more comprehensive analyses of biological, chemical, geological, and hydrographic conditions. Statistical correlation can be improved and costs reduced if subsamples of soil for laboratory toxicity tests, geochemical analyses, and community structure are taken simultaneously from the same grab of the same site.

5.9 Soil toxicity and bioaccumulation tests can be an important tool for making decisions regarding the extent of remedial action necessary for contaminated terrestrial sites.

6. Interferences

6.1 Limitations to the methods described in this guide might arise and thereby influence soil toxicity test results and complicate data interpretation. The following factors should be considered when testing soils:

6.1.1 The alteration of field samples in preparation for laboratory testing (for example, transport, screening, or mixing).

6.1.1.1 Maintaining the integrity of soils during their removal, transport, and testing in the laboratory is extremely difficult. The soil environment is composed of a myriad of microenvironments, redox gradients, and other interacting physicochemical and biological processes. Many of these characteristics influence soil toxicity and the availability of compounds to organisms, microbial degradation, and chemical sorption. Any disruption of this environment complicates interpretations of treatment effects, causative factors, and in situ comparisons.

6.1.1.2 Soils tested at temperatures other than those from the field in which they are collected might affect chemical solubility, partitioning coefficients, and other physical and chemical characteristics.

6.1.2 Interaction among chemicals present in the soil.

6.1.3 The use of laboratory-spiked soils that might not be representative of chemicals associated with soils in the field.

6.1.4 The addition of food to test containers may affect the results of a toxicity test, but it may be necessary to feed the test organisms in long-duration tests (see 11.7, A1.9.1.2, A1.9.5, and A4.10.8).

6.1.5 The addition of solvents to the test containers might obscure the adverse influence of chemicals associated with soil and affect soil quality characteristics.

6.1.6 The natural geochemical properties of test soil collected from the field might not be within the tolerance limits of the test species.

6.1.7 Field-collected soils may contain indigenous organisms including (*1*) the same or closely related species to that being tested and (*2*) microorganisms (for example, bacteria and

molds) and algae species that might grow in or on the soil and test container surfaces.

6.2 Tests may not be applicable with materials that are highly volatile (that is, substances for which the Henry's constant or the air/water partition coefficient is greater than one, or substances for which the vapor pressure exceeds 0.0133 Pa at 25°C) or rapidly transformed biologically or chemically. The dynamics of test material breakdown products should therefore be considered, especially in relation to assumptions of chemical equilibria.

7. Apparatus

7.1 *General Facilities*—The facility should include separate constant temperature areas (chambers) for culturing and testing to reduce the possibility of contamination by test materials and other substances, especially volatile compounds. Culture containers should not be in a room (chamber) in which toxicity tests are conducted, stock solutions or test solutions are prepared, or equipment is cleaned. The facilities should be well ventilated and free of fumes.

7.2 *Equipment and Apparatus*—Equipment and apparatus that contact stock solutions, test solutions, site soils, and test soils, into which test organisms will be placed, should not contain substances that can be leached or dissolved in amounts that affect the test organisms adversely. In addition, equipment and apparatus that contact soils or solutions should be chosen to minimize the sorption of test materials. Glass, Type 316 stainless steel, nylon, high-density polyethylene, polycarbonate, and fluorocarbon plastics should be used whenever possible to minimize leaching, dissolution, and sorption. Copper, brass, lead, galvanized metal, and natural rubber should not be used. Items made of neoprene rubber and other materials not previously mentioned should not be used unless it has been shown that their use will not affect the survival, growth, or reproduction of test organisms adversely.

7.3 *Test and Culture Chambers*—A test or culture chamber is an enclosed space or compartment in which temperature and lighting are controlled (for example, incubator or modified room). The ventilation of chambers, especially test chambers, is desired.

7.3.1 Test and culture chambers usually require continuous lighting (except in the case of the Enchytraeid Reproduction Test). A timing device should be used to provide a light:dark cycle if a photoperiod other than continuous light is used.

7.3.2 Temperature-recording devices should be used to monitor the temperature of test and culture chambers. Both test and culture chambers should be at the same temperature (except in the case of the Enchytraeid Reproduction Test).

7.4 *Culture Containers*—Containers used to culture test organisms should be made of materials that will not affect their survival, growth, or reproduction adversely. Consideration should be given to cleaning and organizational space. The size of culture containers may depend on the species being cultured.

7.5 *Test Containers*—Test containers should be made of materials that minimize the sorption and leaching of test compounds and do not affect the survival, growth, and reproduction of the test organism adversely. Glass is an ideal material.

7.5.1 All test containers used in a soil toxicity test must be identical. The test containers should be covered with a lid to prevent escape of the test organisms and help reduce drying of the test soil.

7.5.2 Species-specific information on test containers and test conditions is given in Annex A1, Annex A3, and Annex A4.

7.6 *Cleaning*—Test containers and equipment and apparatus should be cleaned before use. Items may be cleaned in the following manner: (*1*) scrub thoroughly with a scratch pad to remove visible soil and residue; (*2*) detergent wash; (*3*) water rinse; (*4*) organic solvent wash (for example, acetone); (*5*) acid wash (for example, 10 % concentrated hydrochloric acid); (*6*) tap water rinse; (*7*) rinse at least twice with distilled, deionized, or reagent grade water; and (*8*) dried at room temperature or in a low-temperature (up to 90°C) air-drying oven. Care must be taken to avoid the use of "plastics" that may breakdown in the presence of the solvent used or at prolonged exposures near 90°C. For acceptable items, the following steps may be used alternatively for cleaning: (*1*) scrub thoroughly with a scratch pad to remove visible soil and residue; (*2*) detergent wash; (*3*) water rinse; (*4*) acid wash (for example, 10 % concentrated hydrochloric acid); (*5*) tap water rinse; (*6*) rinse at least twice with distilled, deionized, or reagent grade water; and (*7*) bake in an oven at 350°C. Clean lids should be placed on test containers after the containers have cooled.

7.6.1 A laboratory dish-washing machine may be used to accomplish the detergent wash/water rinse and tap water rinse stages. If a dish-washing machine is used, a neutralizing rinse may be necessary after the acid wash to prevent acid damage to the machine's metal parts.

7.6.2 Many organic solvents leave a film that is insoluble in water. A dichromate-sulfuric acid cleaning solution can generally be used in place of both the organic solvent and the acid, but the solution might leave chromium residues on glass.

7.6.3 Upon completion of a test, all items to be reused should immediately be (*1*) emptied of soil, (*2*) rinsed with water, and (*3*) cleaned by the procedures previously outlined. Test organisms and soil should be disposed of using appropriate procedures (see Guide D 4447).

7.6.4 Test containers should be stored with their lids on to keep them clean.

7.7 *Acceptability*—Before a toxicity test is conducted in new test facilities, it is desirable to conduct a "non-toxicant" test, in which all test containers contain a negative control of artificial or reference soil. Survival, growth, or reproduction of the test species will demonstrate whether the facilities, hydration water, artificial soil, and handling techniques are adequate to result in acceptable species-specific control numbers. The magnitude of the within-chamber and between-chamber variance should also be determined.

8. Safety Precautions

8.1 Many substances pose health risks to humans if adequate precautions are not taken. Information on the chemical and physical properties, toxicity to humans **(18-21)**, and recommended handling procedures **(22-26)** of the test material should be studied and made available to all personnel involved before a test is begun. Contact with the test materials should be avoided.

8.1.1 Many materials can affect humans adversely if precautions are inadequate. Field-collected soils might contain toxic materials, and respiratory exposure and skin contact should be prevented or minimized. As much information as possible should be collected on the history of the site and the potential problems from human exposure. Exposure to workers might be minimized by wearing rubber boots, disposable safety gear, gloves, and a cartridge respirator. Information or directives on necessary precautions should be available from a site safety manager at some sites.

8.1.2 When screening, mixing, or distributing hazardous soils in the laboratory, proper handling procedures might include working (*1*) under a ventilated hood, wearing protective gloves, laboratory coats, aprons, and safety glasses; or (*2*) in a ventilated room, wearing rubber boots, disposable safety gear, gloves, and a full-face bottled air respirator. When initiating toxicity tests in the laboratory, procedures might include wearing appropriate protective gloves, laboratory coats, aprons, and safety glasses and working in a ventilated hood.

8.2 Careful consideration should be given to those chemicals that might biodegrade, transform to more toxic components, volatilize, oxidize, or photolyze during the test period.

8.3 Health and safety precautions and applicable regulations for the disposal of stock solutions, test organisms, and soils should be considered before beginning a test (see Guide D 4447).

8.4 Cleaning of equipment with a volatile solvent such as acetone should be performed only in a well-ventilated area in which no smoking is allowed and no open flame such as a pilot light is present.

8.5 An acidic solution should not be mixed with a hypochlorite solution because hazardous fumes might be produced.

8.6 Concentrated acid should be added to water, not vice versa, to prepare dilute acid solutions. Opening a bottle of concentrated acid and adding concentrated acid to water should be performed only in a fume hood.

8.7 The use of ground fault systems and leak detectors is recommended strongly to help prevent electrical shocks.

9. Soil

9.1 *General*—Before the preparation or collection of soil, an approved, written procedure should be prepared for the handling of soils that might contain unknown quantities of toxic chemicals (see Section 8). All soils should be characterized and have at least the following determined: pH, percent organic matter, cation exchange capacity (CEC), total nitrogen, particle size distribution (percent sand, silt, and clay), and percent water content. In addition, chemical analyses should be performed for compounds suspected of occurring in the particular soil (for example, heavy metals and organics). Toxicological results might provide information directing a more intensive analysis. Soil toxicity testing procedures are detailed in Section 11.

9.2 *Negative Control and Reference Soil*—A negative control soil is used for the following: (*1*) to yield a measure of the acceptability of the test, (*2*) to provide evidence of the health and relative quality of the test organisms, (*3*) to determine the suitability of the test conditions and handling procedures, and (*4*) to provide a basis for interpreting data obtained from the test soils. A reference soil is used to describe the matrix effects of a test. Every test must have a negative control of artificial or reference soil and may also have a reference soil if the negative control is an artificial soil. A reference soil should be collected from the field in a clean area and represent the test soil as much as possible in soil characteristics (for example, percent organic matter, particle size distribution, and pH). This provides a site-specific basis for comparison of toxic and nontoxic conditions. The same conditions, procedures, and organisms must be used with the negative control and reference soil as are used in the other treatments, except that contaminated soil or test materials are not added. In addition, a reference control (artificial or reference soil spiked with a compound with known toxicity at the concentrations(s) used) is desirable.

9.3 *Field Sampling Design*—A site is defined as a delineated tract of land that is being considered as the overall study area, usually from the standpoint of its being potentially affected by xenobiotics. The field collection is often conducted in areas in which little is known concerning contamination or contamination patterns. The object of a qualitative field sampling design is to identify sites that contain potentially toxic conditions that may warrant further study. The collection design might divide the site into sampling units based on habitat or topography to allow for maximum spatial coverage. Sampling stations may be set up within each unit (see 3.2). One sample is collected from each station. The lack of field replication at each station usually precludes statistical comparisons; however, the identification of samples for further study is possible, when survival, growth, or reproduction differ between sampling stations or sampling stations differ from a reference soil. Information on field sampling design is presented by Warren-Hicks, et al **(27)**, Eberhardt and Thomas **(28)**, Gilbert **(29)**, and ISO **(108)**.

9.3.1 If the object of the field sampling design is to test for statistically significant differences in the effects between negative control or reference soils and test soils from several sites or between sampling stations within a single site, a quantitative method is used that requires replicate sampling. The number of field replicates (that is, separate soil samples at a single sampling station) necessary per sampling station is a function of the need for sensitivity or power. A minimum of three field replicates from each station is recommended. These field replicates are each treated as a separate sample in the laboratory, that is, they are not mixed together. The field replicates from a single sampling station might be used (*1*) to test for within-sampling station variability, (*2*) to compare laboratory test procedures, or (*3*) to compare sensitivity among test species.

9.3.2 Sampling stations might be distributed along a known pollution gradient within a site or at random within sampling units. Comparisons can be made between both space and time if the sampling and testing take place during different times of the year.

9.4 *Field-Collected Test Soil*:

9.4.1 *Collection*—A shovel or auger (preferably stainless steel) should be used to collect soil samples (see Section 8). The surface of the location at which the sample is to be

collected should be cleared of debris such as leaves and twigs. If the location is an area of grass or other plants, the plants should be cut to ground level and removed before the sample is collected. The sample should be placed in a thick plastic bag (for example, 4 mil) and taped closed. This bag should then be placed in a second plastic bag, taped closed, and placed in a clean sample container with a lid (for example, plastic pail with O-ring seal). Direct sunlight should be minimized during collection if the chemicals associated with soils include compounds that photolyze readily. All soil samples should be placed in an ice chest and kept cold in the field. Field observations concerning habitat and type of vegetation and measurements such as soil temperature and moisture may be taken in the field.

9.4.2 *Storage*—Soil samples should be utilized as soon as possible in accordance with Test Methods E 1706 stored at 4 ± 2°C for no longer than eight weeks before the start of the test. Freezing and longer storage times might change the soil properties and should be avoided. The soil may be stored in the sample containers in which it was collected in the field. It is desirable to avoid contact with metals and plastics.

9.4.3 *Processing*—The following procedures should be followed if a homogenous sample is needed. The samples should be screened to remove oversize material such as rocks. A 6.30-mm mesh, stainless steel screen may be used. The soil should be mixed after screening (for example, in a stainless steel mixer) to ensure homogeneity (see Section 6). Subsamples of the processed soil should be removed for pH and moisture content determination. Moisture content is determined gravimetrically by drying a subsample for 24 h at 100°C. Information on moisture content is necessary to determine the amount of hydration water to add to the test soils (see A1.9.3). Each replicate is screened, mixed, and treated separately if a quantitative method of field sampling with replicates was used.

9.4.3.1 There may be some instances when an intact core sample needs to be tested, and no processing is therefore necessary.

9.4.4 Qualitative descriptions of the soil may include color, texture, or the presence of roots, leaves, and soil organisms. Monitoring the odor of soil samples should be avoided because of potentially hazardous volatile chemicals (see Section 8).

9.4.5 The natural geochemical properties (for example, pH) of test soil collected from the field should be within the tolerance limits of the test species, or controls for the variable should be run (for example, a pH-adjusted soil). Limits for the test species should be determined in advance (see 10.1).

9.5 *Laboratory-Spiked Test Soil*—Test soil can also be prepared in the laboratory by adding materials such as chemicals or waste mixtures to artificial, reference, or site soils (see 1.4).

9.5.1 Test chemicals should be reagent grade[4] or better, unless technical or other grade material is specifically needed. Before a test is started, the following should be known concerning the test material: (*1*) identity and concentration of major ingredients and impurities; (*2*) water solubility in hydration water, log P_{ow}, and vapor pressure; (*3*) estimated toxicity to the test species and to humans; (*4*) precision and bias of the analytical method at the planned concentrations of the test material, if the test concentrations are to be measured; and (*5*) recommended handling and disposal procedures. Additional information on the fate of the test substance in soil is desirable.

9.5.2 *Stock Solutions*—Test materials to be tested in artificial, reference, or site soil should be dissolved in a solvent (the preferred solvent is water) to form a stock solution. The stock solution itself, or dilutions of it, are then added to the soil. The concentration and stability of the chemical in the stock solution should be determined before beginning the test. The stock solution should be shielded from light both before and during the process of mixing into the soil if the chemical is subject to photolysis. Concentrations of the chemical in the solvent and soil should be monitored before the test begins.

9.5.3 *Non-Water Solvents*—If a solvent other than water is necessary, it should be one that is water-miscible and can be driven off (for example, evaporated), leaving only the test chemical on the soil. Both a solvent control and a negative control soil must be included in the test if a solvent other than water is used. The solvent control must contain the highest concentration of solvent added to the soil and must use solvent from the same batch used to make the stock solution. The same concentration of solvent should be used in all treatments.

9.5.3.1 Acetone is an organic solvent used for preparing stock solutions **(4, 14, 16, 30)** because of its high volatility and ability to dissolve many organic chemicals. Other water-miscible organic solvents, such as methanol or ethanol **(6)**, may be used. Organic solvents may affect total organic carbon levels, introduce toxicity, or alter the geochemical properties of the soil (see 6.1.5). A surfactant should not be used in the preparation of a stock solution because it might affect the bioavailability, form, and toxicity of the test material.

9.5.3.2 If the concentration of solvent is not the same in all test solutions that contain test material, a solvent test should be conducted to determine whether survival, growth, or reproduction of the test organisms are related to the solvent concentration over the range used in the toxicity test. If survival, growth, or reproduction are found to be related to solvent concentration, a soil toxicity test with that species in that amount of solvent is unacceptable if any treatment contained a concentration of solvent in that range.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

9.5.3.3 For compounds insoluble in water and in organic solvents, 10 g of finely ground quartz sand should be mixed with the quantity of test substance to obtain the desired test concentration. This mixture of quartz sand and test substance should be added to the premoistened soil and thoroughly mixed by adding an appropriate amount of deionized water to obtain the moisture required as-described by OECD **(107)**.

9.5.3.4 The survival, growth, or reproduction of the organisms tested in the two controls should be compared if the test contains both a negative control and a solvent control. Only the solvent control may be used for meeting the acceptability of the test and as the basis for the calculation of results if a statistically significant difference in either survival, growth, or reproduction is detected between the two controls. The negative control might provide additional information on the general health of the organisms tested. The data from both controls should be used for meeting the acceptability of the test and as the basis for the calculation of results if no statistically significant difference is detected.

9.5.4 *Test Concentrations*:

9.5.4.1 If the test is intended to allow the calculation of an LC50 or a NOEC, the test concentrations should bracket the predicted LC50 or NOEC. The prediction might be based on the results of a test on the same or a similar test material on the same or a similar species. The LC50 or NOEC of a particular compound may vary, depending on physical and chemical soil characteristics. If a useful prediction is not available, it is desirable to conduct a range-finding test in which the organisms are exposed to a control and three or more concentrations of the test material that differ by a factor of ten.

9.5.4.2 In some situations (for example, regulatory), it might be necessary to determine only (*1*) whether a specific concentration of test material is toxic to the test species or (*2*) whether the LC50 is above or below a specific concentration. When there is interest in a particular concentration, it might be necessary to test only that concentration and not to determine the LC50.

9.5.4.3 If the test is intended to allow the calculation of the ECx (for example, EC_{10}, EC_{50}), the test concentrations should cover the whole range of potential effects. At least three replicates for each concentration and at least six replicates for the controls should be used. The spacing factor may vary, that is, less than two at low concentrations and more than two at high concentrations. If a useful prediction is not available, it is desirable to conduct a range-finding test in which the organisms are exposed to a control and five concentrations of the test material that differ by a factor of ten.

9.5.5 The addition of test materials to soil may be accomplished using various methods such as hand mixing or using a mechanical mixer (see 9.4.3).

9.5.5.1 If tests are repeated, mixing conditions such as the duration and temperature of mixing and time of mixing before the test starts should be kept constant. Care should be taken to ensure that a test material added to a soil is distributed thoroughly and evenly within the soil. The homogeneity of laboratory-dosed material should always be determined prior to testing.

10. Test Organism

10.1 *Species*—Only one species is currently described in this guide (see Annex A1 and Annex A4); however, descriptions of additional species may be included in revisions of this guide. The use of these species is encouraged to increase the comparability of results. The source and type of soil being tested or the type of test to be implemented might dictate the selection of a particular species. The species used should be selected based on (*1*) availability; (*2*) sensitivity to test materials; (*3*) tolerance to parameters such as temperature, pH, and grain size; and (*4*) ease of handling in the laboratory. The species used should be identified using an appropriate taxonomic key.

10.2 *Age*—All organisms should be as uniform as possible in the state of maturity and weight class. The state of maturity or weight class for a particular test species should be chosen so that the sensitivity to test materials is not affected by age, reproduction, or other intrinsic life-cycle factors (see Annex A1 and Annex A4).

10.3 *Source*—All organisms in a test must be from the same source. Organisms may be obtained from laboratory cultures or natural populations from clean areas. Local and state agencies might require collecting permits. Laboratory cultures may be the best source of test species because laboratories can provide organisms whose history, age, and quality are known. State and federal institutions may have available laboratory cultures of test organisms. Commercial suppliers who have laboratory cultures of research and testing organisms may also be a source. It is important to obtain organisms that are of a known species or subspecies and not a mixture. Paragraphs A1.5 and A4.6 contain additional information on possible sources of test organisms.

10.4 *Quality*—Chemical analysis of organisms collected from natural populations is desirable. It may be desirable to analyze for the test materials and other chemicals to which major exposure might have occurred.

10.5 *Care of Brood Stock*—Brood stock should be cared for properly to prevent unnecessary stress (see Annex A1). To maintain organisms in good condition and prevent unnecessary stress, they should not be crowded and should not be subjected to rapid changes in temperature or the quality of culturing medium. Earthworms, but not potworms, should be cultured at the same temperature as that used for testing (see 11.5, A1.9.1.4, A4.5.2, and A4.10.7).

10.6 *Handling*—Test organisms should be handled as little as possible. When handling is necessary, it should be done as gently, carefully, and as quickly as possible. Organisms should be introduced into test soils on the surface so as to evaluate burrowing behavior. Any organisms that touch dry surfaces or are dropped or injured during handling should be discarded.

11. Procedure

11.1 *Experimental Design of Laboratory Experiments*—Decisions concerning the various aspects of experimental design, such as the number of treatments and number of test containers and test organisms per container, should be based on the purpose of the test and the type of procedure that is to be used to calculate results (see Section 14). A test intended to

allow the calculation of a specific endpoint such as an LC50 should consist of a negative control, a solvent control, if necessary, and several test concentrations (see 9.5.3).

11.1.1 The primary focus of the experimental test design and statistical analysis of the data is the experimental unit, which is defined as the smallest physical entity to which treatments can be assigned independently **(31)**. The test container is the experimental unit (see 7.5). As the number of test containers per treatment increases, the number of degrees of freedom increases, and therefore the width of the fiducial interval on a point estimate, such as an LC50, decreases, and the power of a significance test increases (see Section 14). Because of factors that might affect the results within test containers and therefore the results of the test, (*1*) all test containers must be treated as similarly as possible, for example, temperature and lighting, and (*2*) each test container must be treated physically as a separate entity. The assignment of test organisms to test containers must be randomized, and test containers must be assigned randomly to individual test chamber locations.

11.2 *Soil Into Test Containers*—The day before the toxicity test is started (Day − 1), the soil to be tested, negative control, and reference soil (if used) are mixed, the moisture level is adjusted with hydration water, and the soils are placed into test containers. Paragraph A1.9.3 contains information on the hydration of test soils. If large interstitial spaces of air occur in the soil matrix, these spaces should be removed by pressing in the soil with a suitable utensil, for example, a spatula (see 7.2), while trying not to compact the soil. The minimum amount of soil to mix and hydrate should be enough for three replicates, a moisture sample, a pH sample, and to account for soil adhering to the sides of the mixing chamber. This mixed and hydrated soil is called a batch. Extra batch soil may be mixed and hydrated if a sample is to be removed for chemical analysis or for any other purpose. Site soil has been mixed previously during processing.

11.2.1 *Site Soil Sampler*—From each sample collected at a field station, soil sufficient for at least three replicates is hydrated with water, and replicates are placed into test containers (see Annex A1 and Annex A4).

11.2.2 *Test Soils Prepared for a Concentration Series*—If site soil and artificial or reference soil are to be mixed in a concentration series, each concentration (treatment) is prepared as a batch from which replicates are placed into test containers. If site, reference, or artificial (see Annex A2) soil is to be spiked with chemicals, each concentration is prepared as a batch, and replicates are placed into test containers.

11.2.3 The test containers with soil are covered with a lid containing a very small hole to allow for air movement. The test containers are then placed into the test chamber, until the next day, to (*1*) allow the test containers to temperature equilibrate and (*2*) allow time for the test material to equilibrate with the soil. Each test container must contain the same amount of soil (specified in Annex A1) determined on a dry weight basis.

11.3 *Introduction of Test Organisms*—Test organisms are placed into the test containers after the overnight equilibration; this constitutes the beginning of the test (Day 0). The test organisms are placed on the surface of the soil and allowed to burrow because a lack of burrowing is considered a response possibly due to the presence of toxic compounds **(6)**.

11.4 *Duration of Test*—The test begins when test organisms are first placed in the test containers and continues for the duration specified in the experimental design for a specific test organism.

11.5 *Temperature*—In toxicity tests with *E. fetida* in artificial soil with 2-chloroacetamide and benomyl, Heimbach and Edwards **(32)** found that temperature variations between 10 and 26°C had little influence on the toxicity of the chemicals. In the case of *E. albidus*, any temperature higher than 22°C should be avoided since reproduction can be affected. The test temperature depends on the species used (see Annex A1 and Annex A4). Other temperatures may be used to study the effect of temperature on the survival, growth, or reproduction of test organisms and contaminant-related properties (for example, bioavailability).

11.6 *Test Measurements*:

11.6.1 Temperature should be monitored for the duration of the test. A continuous temperature recorder (or a continuous temperature/humidity recorder) with a seven-day chart can be placed in the test chamber and changed as necessary.

11.6.2 A rough measurement of the total biomass of test organisms per test container should be obtained at the beginning of the test. A rough measurement consists of weighing the worms after first removing any large fragments of bedding that may be adhering to them (see A1.7 and A1.7.1).

11.6.2.1 If weight loss is used as an endpoint, an accurate measurement of weight must be taken of the total biomass of test organisms per test container at the beginning and end of the test. The worms should be purged of their gut contents before weighing by placing them in petri dishes with wet filter paper. Bedding should be rinsed from the worms with test water before placing the worms in petri plates. Before weighing the worms, excess surface water may be removed by placing the worms between layers of an absorbent towel. It is very important not to dry the surface of the worms, and consideration should be given to whether this step might stress the worms unduly. Researchers have commonly used 24 h **(7, 12, 33)** or 48 h **(34, 35)** for a purging time period. Although Stafford and McGrath **(35)** provided some evidence that some soil may still remain in the gut after 48 h, it is recommended that 24 h be used as a purging time. An excessively long period of starvation prior to initiating a lengthy test during which food is not added (see 11.7) may stress the test organisms.

11.6.2.2 Richards and Ireland **(36)** suggest that longer periods of starvation may result in the depuration of heavy metals from earthworm tissue. These factors need to be considered if bioaccumulation studies are to be performed, and an elimination study should be undertaken to determine the effect of purging on the concentration of the target compounds in the earthworms.

11.6.3 pH should be measured (see A1.11.1) at the beginning of the test in subsamples taken from the batch preparations and at the end of the test in subsamples from replicates of the various concentrations.

11.6.4 Percent moisture may be measured (see A1.11.2) at the beginning and end of the test from subsamples, as noted in 11.6.3.

11.6.5 Salinity should be measured (see A3.7) at the beginning and end of the test (except in the case of the Enchytraeid Reproduction Test). This may be done in subsamples as noted in 11.6.3.

11.7 *Food*—It is recommended that food not be added to the test containers because it may affect the results of the test. In studies of longer duration, that is, over 28 days, the use of food may have to be reevaluated (see A1.9.1.2, A1.9.5, and A4.10.8).

11.8 *Light*—To maximize exposure, continuous lighting **(14, 37)** using either a fluorescent or an incandescent light source must be used for testing. A minimum intensity of 37 fc (400 lux) is recommended for testing **(37)**. In the case of the Enchytraeid Reproduction Test, a controlled light-dark cycle of long-day conditions (preferabley 16 to 8 h at 400 to 800 lux in the area of the test vessels) is desirable.

11.9 *Biological Data*—Effects indicating the toxicity of a test soil include mortality and may include sublethal effects on growth, behavior, reproduction, and physiological processes, as well as observations on external pathological changes, for example, segmental constrictions, lesions, or stiffness (see A1.10 and A4.10.13.2). Toxicity test containers may be observed on a weekly basis or only at the end of the test. Test soil and organisms are emptied onto a flat surface, and the organisms are removed and evaluated, at the end of the exposure period.

11.10 *Chemical Analyses*:

11.10.1 *Field-Collected Soils*—Soil samples for laboratory testing should be collected from the same grab as for chemical analysis. A subsample from the same grab may be used for faunal analyses.

11.10.2 *Artificial Soil and Field-Collected Soils Spiked in the Laboratory*—Measurement of the concentration of test materials in the batches of test soil is desirable at the beginning of the experiment. Chemical analyses at several concentrations of soil from the test containers may be made at the end of the test. To monitor changes in soil chemistry during the course of the experiment, separate test containers may be set up (including test organisms) and sampled as necessary or practical over the duration of the experiment. The measurement of test materials degradation products might also be desirable.

11.10.3 *Tissue Analysis*—Contaminant bioavailability is indicated by the chemical concentrations accumulated in earthworm tissues (see A3.8.3).

12. Analytical Methodology

12.1 Chemical and physical data for soil and tissue material should be obtained using appropriate ASTM International standards whenever possible. For those measurements for which ASTM International standards do not exist or are not sufficiently sensitive, methods should be obtained from other sources, for example, EPA **(38)**.

12.2 Concentrations should be measured for (*1*) chemicals in batches of soil, (*2*) test materials in stock solutions, and (*3*) chemicals in test containers. In addition, measurements for the presence of an apparently evaporated organic solvent may be desirable.

12.2.1 If samples of stock solutions or test soils are not to be analyzed immediately, they should be handled and stored appropriately (see 9.4.2).

12.3 Methods used for analyzing test organisms for chemicals of concern should be obtained from appropriate sources **(39)**.

12.4 The precision and bias of each analytical method used should be determined in an appropriate matrix, that is, soil, water, or tissue. When appropriate, reagent blanks, recoveries, and standards should be included when samples are analyzed.

13. Acceptability of Test

13.1 A soil toxicity or bioaccumulation test should be considered unacceptable if one or more of the following situations occurred.

13.1.1 Continuous lighting had not been used during the test, if soil exposures were intended to be maximized (see 11.8), unless performing the bioaccumulation assay test variation with Bermuda grass (see A3.10) or the Enchytraeid Reproduction Test (see A4.10.7).

13.1.2 All test containers were not identical (see 7.5 and 11.1).

13.1.3 Test organisms were not cultured at the same temperature as that used for testing (see 7.3.2, 10.5, and 11.5) except in the case of the Enchytraeid Reproduction Test.

13.1.4 The natural geochemical properties of test soil collected from the field was not within the tolerance limits of the test species (see 9.4.5).

13.1.5 Appropriate negative and solvent controls were not included in the test (see 9.2 and 9.5.3).

13.1.6 The concentration of solvent in the range used affected the survival, growth, or reproduction of the test organisms (see 9.5.3.2).

13.1.7 All animals in the test population were not obtained from the same source, were not all of the same species, or were not of acceptable quality (see Section 10 and A4.10.10).

13.1.8 Treatments were not assigned randomly to individual test chamber locations, and individual test organisms were not assigned randomly to test containers (see 11.1.1).

13.1.9 Each test chamber did not contain the same amount of soil, determined on a dry weight basis (see 11.2).

13.1.10 The temperature was not within the acceptable range (see A1.9.1.4, A3.7, and A4.10.7).

13.1.11 The negative control soil organisms did not survive, grow, or reproduce as required for the test species (see 9.2, Annex A2, and Annex A4).

14. Calculation of Results

14.1 The calculation procedures and interpretation of the results should be appropriate to the experimental design. Procedures used to calculate the results of toxicity tests can be divided into two categories: those that test hypotheses and those that provide point estimates. No procedure should be used without careful consideration of (*1*) the advantages and

disadvantages of various alternative procedures and (*2*) appropriate preliminary tests, such as those for outliers and heterogeneity.

14.2 The LC50 or EC50 and its 95 % fiducial limits should be calculated (when appropriate) for each set of data on the basis of the measured initial concentrations of test material, if available, or the calculated initial concentrations. If other LC or ECs are calculated, their 95 % fiducial limits should also be calculated.

14.3 Most toxicity tests produce quantal data, that is, counts of the number of responses in two mutually exclusive categories, such as alive or dead. A variety of methods **(40-43)** can be used to calculate an LC50 or EC50 and 95 % fiducial limits from a set of quantal data that is distributed binomially and contains two or more concentrations at which the percent dead or effected is between 0 and 100, but the most widely used are the probit, moving average, Spearman-Karber, and Litchfield-Wilcoxon methods. The method used should take into account appropriately the number of test organisms per container. The binomial test can also be used to obtain statistically sound information concerning the LC50 or EC50 even when fewer than two concentrations kill or affect between 0 and 100 %. The binomial test provides a range within which the LC50 or EC50 should lie. In a case in which few data are available, the geometric mean (the root of the multiplication of LC0 and LC100) or a nonlinear interpolation may be used to determine the LC50 or EC50.

14.4 When samples from field stations are replicated independently, the effects at those stations can be compared statistically by *t*-tests, analysis of variance (ANOVA), or regression-type analysis. The ANOVA is used to determine whether any of the observed differences among the samples (or concentrations) are statistically significant. This is a test of the null hypothesis that no differences exist in the effects among the samples (or concentrations) and the control. If the *F*-test is not statistically significant ($P > 0.05$), it can be concluded that the effects observed in the test material treatments (or field stations) were not large enough to be detected as statistically significant by the experimental design and hypothesis test used. Non-rejection does not mean that the null hypothesis is true. The NOEC based on this end point is then taken to be the highest test concentration tested **(44)**. The amount of effect that occurred at this concentration should be considered.

14.5 All exposure concentration effects (or field stations) can be compared with the control effects by using mean separation techniques, orthagonal contrasts, Fisher's methods, Dunnett's procedure, or Williams' method. The lowest concentration for which the difference in observed effect exceeds the statistically significant difference is defined as the LOEC for that end point. The highest concentration for which the difference in effect is not greater than the statistically significant difference is defined as the NOEC for that end point.

14.6 Bioaccumulation test results are reported as the magnitude of chemical concentration above either the Day 0 tissue baseline analysis or the Day 28 tissues from the negative control or reference soil (that is, 2×, 5×, 10×) (see A3.9). Other approaches for evaluating data include kinetics studies with estimate uptake, depuration rates, and time to steady state, lipid normalization and normalizing soil concentrations of non-ionic organics to TOC (see Guide E 1688). Analysis of field collected organisms is also an option.

14.7 Three designs are possible for the test performance (the concentrations should be spaced by a factor not exceeding two): *(1)* For determination of the NOEC, at least five concentrations in a geometric series should be used. Four replicates for each treatment plus eight controls are recommended. *(2)* For determination of the ECx (for example, EC_{10}, EC_{50}), twelve concentrations should be used. Two replicates for each treatment and six control replicates are recommended. The spacing factor may vary, that is, less than two at low concentrations and more than two at high concentrations. *(3)* For the mixed approach, eight concentrations in a geometric series should be used. Four replicates for each treatment plus eight controls are recommended. This combined approach allows for determination of both the NOEC and ECx.

14.8 The ECx approach can be used for the Enchytraeidae reproduction test described in Annex A4. To compute any ECx value, the per-treatment means are used for regression analysis after an appropriate dose-response function has been obtained. An ECx is calculated by inserting a value corresponding to x % of the control mean into the equation obtained by regression analysis. The 95 % confidence limits are calculated according to Fieller **(122)**. Alternatively, the results can be expressed as percentages of inhibition relative to the control. In these cases, the normal (logistic) sigmoid curve can often be fitted to the results by use of the probit regression procedure **(121)**. But if the hormesis phenomenon has been observed, probit analysis should be replaced, for example, by a four-parameter logistic or Weibull function fitted by a nonlinear regression procedure.

15. Report

15.1 Include the following information, either directly or by reference to available documents, in the record of the results of an acceptable soil toxicity test:

15.1.1 Name of the test and investigator, name and location of the laboratory, and dates of the start and end of the test.

15.1.2 Source of the negative control, reference, or test soil.

15.1.3 Method of the collection, handling, shipping, storage, and disposal of soil.

15.1.4 Source of the test material; lot number, if applicable; composition (identities and concentrations of major ingredients and impurities, if known); known chemical and physical properties; and, if necessary, application of the test compound.

15.1.5 Identity and concentration of any solvent used.

15.1.6 Source and quality of hydration and test water.

15.1.7 Source, history, and reproductive status of the test organisms; scientific name, name of person who identified the test organism, and taxonomic key used; culture procedures and any observed diseases, unusual appearance, or treatments; source of culture and date the culture stock was obtained; and biomass of test organism per test container.

15.1.8 Source and composition of food, concentrations of test material and other chemicals, procedure used to prepare food, and feeding methods and frequency.

15.1.9 Description of the experimental design and test chambers; weight (dry weight basis) of the test soil in each test container; amount of hydration water added to the test soil;

type and intensity of lighting in the test chamber; number of test containers and number of test organisms per container and per treatment; date and time the test started and ended; temperature measurements during the test; pH values of test soils at the start and end of the test; and any other measurements taken.

15.1.10 Methods used for, and results (with standard deviations or fiducial limits) of, the physical and chemical analyses of site soil, test soil, and stock solutions.

15.1.11 Definition(s) of the effects used to calculate LC50 or EC50s, biological endpoints for tests, and a summary of general observations of other effects.

15.1.12 A table of the biological data for each test container for each treatment, including the control(s) in sufficient detail to allow independent statistical analysis.

15.1.13 Methods used for, and results of, the statistical analyses of data.

15.1.14 Summary of general observations on other effects or symptoms.

15.1.15 Anything unusual concerning the test, any deviation from these procedures, and any other relevant information.

15.1.16 Published reports should contain enough information to identify clearly the methodology used and the quality of the results.

16. Keywords

16.1 bioaccumulation tests; earthworm; potworms; soil toxicity

ANNEXES

(Mandatory Information)

A1. *EISENIA FETIDA*

A1.1 *Significance—Eisenia fetida* (Savigny, 1826), Oligochaeta, has many desirable characteristics for a test species: (*1*) it has a short generation time **(45)**; (*2*) it reproduces prodigiously **(46)**; (*3*) it is collected easily from natural sources or cultured in the laboratory **(2, 37, 47, 48)**; and (*4*) data on its survival, growth, and reproduction can be obtained in toxicity tests **(47, 49-54)**. Stafford, et al **(55)** indicated that *E. fetida* was the most sensitive species, of those examined, for indicating heavy metal availability from soils and dredged sediments. *E. fetida* has been used successfully as a laboratory test organism in many testing mediums, for example, artificial soil **(3)**, contaminated field soils **(5, 56)**, activated sludge **(50)**, sediment **(57)**, and cow manure **(16)**.

A1.2 *Life History*—The life-cycle of *E. fetida* can be divided into three distinct phases, according to Jefferies and Audsley **(58)**: (*1*) the cocoon phase, consisting of an egg cocoon that can produce from one to eleven hatchlings under laboratory conditions **(59)**; (*2*) the young (immature) phase, during which the hatchlings grow physically but cannot produce cocoons; and (*3*) the adult (mature) phase, which is reached when the worms become capable of producing cocoons. Adult worms may still grow physically. Tomlin and Miller **(59)** report a life-cycle for *E. fetida* to vary from a mean of 51.5 days at 25°C to more than 166 days at 13°C, that is, from freshly deposited cocoon through clitellate worm and deposition of the next generation of cocoons. Reynolds **(60)** indicates that *E. fetida* has a maximum life expectancy of 4 to 5 years, although between 1 and 2 years is more usual.

A1.2.1 *E. fetida* is an epigeic species, that is, they live and feed on the surface **(1, 61)** that rarely inhabits agricultural soils but is found in compost piles, manure piles, and other disturbed sites rich in organic matter **(11)**. The rate of soil consumption in the laboratory for *E. fetida* has been estimated at 16 mg soil/individual/day (300 mg, live weight individuals) **(5)**.

A1.2.2 The specific sources of nutrition for *E. fetida* are not well understood, but Morgan **(62)** found that *E. fetida* was capable of using both the microorganisms found in organic wastes and simple nutrients for growth. Worms grew well on pure cultures of four species of fungi and on low concentrations of glucose and sucrose, but they died or lost weight on pure cultures of various bacteria and protozoa species. Worms confined with a single food source may have been exposed to the buildup of toxic metabolites produced by the microorganisms. More work needs to be performed in this area.

A1.2.2.1 Worms digest the microorganisms from ingested soil and organic debris, which illustrates their interactions with the soil environment. This occurs independently of whether mineral matter or fibrous organic material was ingested. Approximately 2.5 h were required at 25°C for passage of ingesta from mouth to anus for *E. fetida* **(63)**.

A1.2.3 Although an increase in temperature within the range from 13 to 25°C reduces the amount of time needed for a life cycle, Tomlin and Miller **(59)** report that an increase in temperature within this range reduces the number of hatchlings per cocoon.

A1.3 *Taxonomy*—The taxonomic status of what Bouché **(61)** calls the *E. fetida* complex is unclear in the literature. Some authors consider this complex to consist of two subspecies, *E. fetida fetida* and *E. fetida andrei*, while other authors consider the complex to consist of two separate species, *E. fetida* and *Eisenia andrei*. This guide chooses to use the subspecies designations. The dorsal surface of *E. f. andrei* is uniformly reddish, while *E. f. fetida* is striped or banded. Fender **(64)** (classifying the two earthworms as different species instead of subspecies) describes *E. fetida* as having pigment covering only the center two thirds or so of the dorsal half of each segment, presenting a strongly banded appearance. He describes *E. andrei* as having pigment covering at least nine

tenths of the length of each segment dorsally, giving it a nearly solid color. He indicates that the taxonomy in the literature is submerged in that of "*E. fetida*," making it unclear which of the two forms is being discussed.

A1.3.1 Roch, et al **(65)** and Valembois, et al **(66)** demonstrated biochemical differences between the two forms. Oien and Stenersen **(67)** and Jaenike **(68)** conducted electrophoretic work that led them to consider the two forms as separate species, and Sheppard **(69)** added research indicating that ecological differences exist between the two forms. It is important to know which form is being used as a test organism for these reasons.

A1.3.2 Bouché **(61)** states that the *andrei*form is relatively homogeneous, while *fetida* may be multispecific. It is recommended that the *andrei* form be used as the test organism, that is, *E. f. andrei*.

A1.4 *Culture of Test Organisms*—The following culture procedures are adapted from Edwards **(37)** and Greene, et al **(17)**. *E. fetida* can be reared in a bedding of sphagnum (*Sphagnum*) peat moss pH adjusted to 7.0 with pure calcium carbonate and hydrated with test water, for example, distilled, deionized, or reverse osmosis. Plastic trays measuring approximately 34 by 28 by 14 cm can hold 700 g (dry weight) of peat moss hydrated with approximately 2300 mL of reagent water. The trays need to be covered, for example, with plastic, to prevent drying. Moisture should be monitored on a weekly basis. The trays should be maintained so that there is no standing water in the bottom of the trays and so that the surface of the bedding is not dry. Placing a piece of material such as plywood over the plastic will keep it in place. The trays are held under continuous lighting at 22 ± 3°C (see A1.9.1.4).

A1.4.1 *E. fetida* have been cultured with a variety of foods, for example: (*1*) cellulose and activated sludge **(70)**, (*2*) dairy waste sludge cake **(71)**, (*3*) horse manure **(7)**, (*4*) activated sludge and horse manure **(72)**, and (*5*) commercial alfalfa pellets (*Medicago sativa*) **(56)**. Alfalfa pellets saturated with test water (at a ratio of approximately 1 g of dry pellets per 2 mL test water) and aged for two weeks in a covered container are consumed readily by *E. fetida*. Alfalfa pellets may be less likely to contain unknown compounds than the other feeds and are therefore recommended.

A1.4.1.1 The worms should be fed once or twice per week, depending on the number of individuals in a tray. Any remaining food is removed and discarded at feeding time. The bedding is then turned by hand to inspect the general condition of the worms and the bedding. If any dead worms are noticed, they should be removed. The tray should be set aside for more frequent evaluation, or it should be discarded, if many dead or stressed-appearing worms are found. Test water is added, and the bedding is turned again, if the bedding needs more moisture. Food is sprinkled over the surface of the bedding in an amount that has been determined will be consumed by the next feeding time.

A1.4.1.2 Some of the pests associated with the culture of worms are fungus gnats, soil mites, Collembola (small insects, commonly called springtails, which are abundant in moist leaf mold, soil, and rotten wood), and enchytraeids (small, white worms belonging to the Class Oligochaeta). None of these pests in low numbers appears to be a problem for the culture of healthy worms. Gnats are seasonal and are mostly a nuisance for the caretaker of the worms. Large numbers of mites and enchytraeids appear to compete for food with the worms, and mites have been observed on dead or dying worms. Biocides are not used for the control of pests because of their potential effect on earthworm health or testing sensitivity. The control of pests consists of removal by hand or by disposal of infected trays. Different geographical regions may have their own distinct types of pests.

A1.4.2 Earthworms should be cultured so they are not stressed unnecessarily. To maintain *E. fetida* in good condition and prevent unnecessary stress, the cultures should be kept at a constant temperature, the pH should be maintained near 7.0, feeding should be on a regular schedule, the moisture level of the bedding should remain adequate as described in A1.4, and crowding (see A1.4.2.1) should be prevented.

A1.4.2.1 Neuhauser, et al **(45)** calculated carrying capacities for *E. fetida*, in a volume of 300 cm^3 with a surface area of 78 cm^2, to range from approximately 6 to greater than 23 g of worm, depending on the type of food source and substrate. This is approximately 0.02 to 0.08 g of worm/cm^3 of substrate. The number of worms that a tray holds is a function of the size and age of the worms. Adult worms have distinct, fully developed clitella and weigh a minimum of approximately 300 mg. Sub-adult worms have visible, but not fully developed, clitella and are approximately 150 to 300 mg in weight. Juvenile (young) worms do not have clitella and are usually less than 150 mg in weight. For optimal reproduction, it is recommended that the trays containing 9000 cm^3 of bedding hold a maximum of 245 g of worm, that is, 0.03 g/cm^3. For example, 350 adult worms weighing 700 mg each would be equal to 0.03 g/cm^3. To reduce the population of worms in a crowded tray, first prepare a new tray of bedding. Half of this new bedding is removed and placed on a piece of plastic sheeting. Half of the bedding containing a portion of worms from the crowded tray is placed into the new tray, and the bedding is mixed by hand. The half tray of new bedding on the plastic sheet is then added to the old tray of bedding and mixed.

A1.4.3 A tray will periodically need to have its bedding changed, even if it is not overcrowded. Prepare a new tray of bedding, and place the contents of the old tray of bedding on top of the new bedding. Allow this tray to sit uncovered in the continuously lighted culture chamber for two days, and allow the worms to burrow into the new bedding. Remove the old bedding from the top of the new bedding and discard. This procedure does not recover the cocoons, and some of the worms will still be in the old bedding.

A1.4.3.1 If it is critical to save each individual worm and cocoon, old bedding that needs changing can be spread onto a sheet of plastic, and every worm and cocoon can be picked by hand and placed into a new tray of bedding. Cocoons should be buried in the new bedding, but worms can be placed on the surface of the new bedding and allowed to burrow.

A1.5 *Obtaining Brood Stock—E. fetida* has been reared on earthworm farms and sold in every Canadian province and American state for fish bait **(60)**. However, bait farms may

contain mixtures of *E. f. andrei* and *E. f. fetida*. Reynolds **(60)** and Fender **(64)** report that *E. fetida* can be found in manure piles and usually not far from human activity. Fender **(64)** (classifying the two earthworms as different species instead of subspecies) states further that if the two earthworms are found in the same manure pile, *E. andrei* is usually found in dryer areas than *E. fetida* and is often most abundant in or below the soil contact region. Starter cultures might also be obtained from various institutions, laboratories, and biological firms, although it is important to ensure a pure culture. Field-collected *E. fetida* should be identified using adult worms. The taxonomic key of Fender **(64)** may be useful for this purpose.

A1.6 *Handling—E. fetida* should be handled as little as possible. When handling is necessary, it should be done as gently, carefully, and quickly as possible, so that the worms are not stressed unnecessarily. Any worms that are dropped or injured during handling should be discarded.

A1.7 *Age*—Tests with *E. fetida* should be started with sexually mature, fully clitellate adults **(5, 7, 17, 47, 56)**. The biomass of earthworms in each test container should be obtained.

A1.7.1 Worms are selected randomly and removed by hand from a culture tray and weighed in groups of ten (see A1.9.1) for each test container. Worms are purged of their gut contents prior to weighing only if weight loss is used as an endpoint (see 11.6.2.1).

A1.8 *Acclimation*—It is recommended that the test organisms be cultured and tested at the same temperature (see 11.5, A1.4, and A1.9.1.4) so that a period of acclimation to temperature is not necessary.

A1.9 *Toxicity Test Specifications:*

A1.9.1 *Experimental Design*—Decisions concerning the various aspects of experimental design, such as the number of concentrations and number of test containers and earthworms per concentration, should be based on the purpose of the test and the procedure used to calculate the results.

A1.9.1.1 Neuhauser, et al **(4)** used a minimum of five concentrations, with four replicates for each test concentration and ten worms per test container, for a definitive test in artificial soil. Each test container consisted of a glass dish 6.5 cm in height and 12.5 cm in diameter (0.8 L) that contained 400 g (dry weight) of test soil. Haque and Ebing **(47)** used five concentrations, with three replicates for each concentration and six worms per container, for a definitive test in artificial soil. Test containers were 1-L glass jars and held 500 g (dry weight) of test soil. Greene, et al **(17)** recommended a minimum of five concentrations, with three replicates per concentration and ten worms per container, for a definitive test in site soil mixed with artificial soil to make a "dilution" series. Test containers were 473-mL glass jars that held 200 g (dry weight) of test soil.

(*1*) It is recommended that a minimum of five concentrations, with a minimum of three replicates per concentration, be used for a definitive test. Ten worms per container is recommended.

(*2*) Using the data on the rate of consumption of soil given in A1.2.1 and assuming that a 600-mg individual would consume twice as much soil as a 300-mg individual, a 28-day test with ten worms weighing 600 mg each would consume only 9 g of soil. High stocking densities, that is, gram earthworm/gram soil, may increase the possibility that earthworms would ingest soil more than once, which may affect the uptake (and therefore toxicity) of compounds **(5)**. Under high stocking densities, the death of an individual earthworm during a test may also be more likely to influence the remaining individuals adversely. It is recommended that each test container hold 200 g (dry weight) of test soil. This amount is well above the potential amount that ten earthworms would process in 28 days. If hazardous waste soils are being evaluated in a laboratory setting, it is important to try to reduce the amount of soil being transported from field to laboratory and the amount of waste generated by the laboratory, both from an economical and environmental viewpoint.

A1.9.1.2 The duration of the test, with mortality as the endpoint, is typically 14 days **(15, 56, 73, 74)**, with an evaluation at seven days being optional. Tests investigating the bioaccumulation of xenobiotics in field-collected soils have been conducted for 56 days **(5)** without the addition of food, but consideration should be made for the possible effect of a lack of food for time periods of this length (see A1.9.1.3 and A1.9.5).

(*1*) Loss in body weight and behavioral and morphological endpoints such as coiling, segmental swellings, segmental constrictions, lesions, rigidness, and flaccidness can be used successfully in toxicity testing **(75-77)**.

A1.9.1.3 Growth and reproduction can be used as biological endpoints in tests with *E. fetida* of longer duration, for example, 140 days **(51)**. The use of food must be considered in long-term growth and reproduction studies (see A1.9.1.2 and A1.9.5). The growth of young worms, rate of clitellum development, number of cocoons produced, cocoon mass, number of hatchlings per cocoon, and biomass of hatchlings have all been used as endpoints in research by Reinecke and Venter **(16)**, Malecki, et al **(51)**, Van Gestel, et al **(54)**, and Venter and Reinecke **(78)** with xenobiotics. The importance of controlling environmental factors such as pH, temperature, and moisture content in growth and reproduction tests has been demonstrated by Van Gestel, et al **(79)**.

A1.9.1.4 Although Heimbach and Edwards **(32)** tested *E. fetida* successfully within the range from 10 to 26°C (see 11.5), the majority of the testing with *E. fetida* has been conducted within the temperature range from 18 to 25°C **(4, 14, 30, 47, 49, 56, 74, 75)**. Van Gestel, et al **(79)** report that a temperature range from 20 to 25°C is optimal for *E. fetida*. Kaplan, et al **(72)** report that *E. fetida* survived best over the temperature range from 20 to 29°C and that mortality was produced at 5 and 33°C. A temperature range from 19 to 25°C is highly recommended for testing, but the temperature range must not fall below 10°C **(32)** or above 29°C **(72)**. (See Table A1.1.)

A1.9.1.5 *E. fetida* has been tested under continuous lighting and with a photoperiod of 12 h light and 12 h dark. A continuous lighting regimen is recommended in order to help keep the photosensitive earthworms burrowing. When measured, lighting intensity has been reported for toxicological

TABLE A1.1 Test Specifications for the 14-Day *Eisenia fetida* Toxicity Test

Test Duration	14 days
Biological endpoint	Mortality
Temperature	19–25°C
Photoperiod	24 h/400 to 1080 lx
Test containers	473-mL glass jars

testing with *E. fetida* from 37 to 100 fc (400 to 1080 lx). A minimum of 37 fc is recommended for testing (see Table A1.1).

A1.9.2 *Test Containers*—Glass testing containers have been used by most researchers with *E. fetida*. Glass, 473-mL canning jars are convenient and have been used successfully with 200 g (dry weight) of test soil **(17)**. Canning jar lids may be used for a cover and held in place with the screw ring. A small (1- to 2-mm) hole should be placed in the center of the lid to allow for air exchange.

A1.9.3 *Day Prior (Day − 1) to Initiation of Test*:

A1.9.3.1 Test soils are hydrated and mixed well into batches, separated into replicates, and placed into test containers that are placed into the test chamber for overnight equilibration (see 11.2). No standing water should be present in the test containers. If a site, reference, or artificial soil is spiked with chemicals or compounds in solution, the solution is used as part of the hydration water.

(*1*) *Tests With Whole (100 %) Site or Reference Soil*—If the negative control is artificial soil, it is hydrated to 35 to 45 % of its dry weight, for example, 660 g (dry weight) would be hydrated with 231 to 297 mL of water. The site and reference soils are also hydrated to 35 to 45 % of their dry weight. Since most soils collected in the field contain some moisture, this moisture content is obtained and used for determining how much additional water to add to the soils to gain a hydration level of 35 to 45 %.

(*a*) Hydrating soils to a standard level is problematic. Because of the variation in water holding capacity (influenced by factors such as soil texture, structure, and organic matter content) between soils, one soil may appear very wet and even have standing water on the surface after hydration to 45 % of its dry weight, and another soil may appear considerably dryer after the same level of hydration. An alternative method for hydrating site and reference soils is to use the artificial soil when hydrated at 45 % of its dry weight as a standard. The site and reference soils can be hydrated to a level approximating the appearance of the artificial soil. Another alternative is to measure the water holding capacity of the soil and then hydrate the soil to 75 % of the water holding capacity value **(17)**. Measuring the water potential **(80)**, for example, using a tensiometer, of the soil may prove to be a better method of hydrating soils. The water potential of artificial soil hydrated to 35 to 45 % of its dry weight could be determined. Soils could be hydrated to the water potential value obtained for the artificial soil using this as a standard. Some variation in the moisture content between soils being evaluated may be acceptable based on the results of the research noted immediately below. Studies by Stafford and Edwards **(5)** with *Eisenia fetida* and five different soils found that a variation in moisture content of 25 to 45 % (presumably moisture content on a wet weight basis) made little difference in the rate of weight loss in the earthworms. Using 2-Chloroacetamide and Benomyl in artificial soil with *Eisenia fetida*, Heimbach and Edwards **(32)** found that changes in the water content of the artificial soil from 17.5 to 51 % of its dry weight had little influence on the toxicity of the chemicals.

(*b*) A sediment can be defined as a naturally occurring particulate material that has been transported and deposited at the bottom of a body of water, or an experimentally prepared substrate within which the test organisms can interact (see Guide E 1383). The definition of a soil as defined within this guide (see Section 3) indicates that a soil is not usually covered by water. It is sometimes difficult to distinguish between a soil and a sediment that has been dried out or deposited on dry land. Although earthworms can survive in a sediment for the duration of the test if the dissolved oxygen content is adequate, earthworms are not recommended for the evaluation of sediments, that is, sediments taken from below a body of water.

(*2*) *Tests With Site Soil Diluted With Artificial Soil*—The artificial soil portion of each concentration is hydrated to 35 to 45 % of its dry weight. The site soil portion of each concentration is hydrated as in (*1*) above. These two portions are then mixed together to form the batch for each concentration from which the replicates are taken.

(*3*) *Tests With Artificial Soil Spiked With Compounds*—If a series of concentrations is prepared by spiking artificial soil with solutions of compounds, the artificial soil is hydrated to 35 to 45 % of its dry weight with test water and the chemical solution combined to make the necessary amount of hydration. If a series of concentrations is prepared by spiking artificial soil with dry chemicals, the chemical is first mixed into the artificial soil very well. The artificial soil is then hydrated with test water, and the batch is mixed again very well before being separated into replicates.

A1.9.4 Earthworms are introduced to the test containers the day after the equilibration period (Day 0). Groups of ten earthworms must be assigned randomly to the individual test containers. Earthworms are removed from the culture trays and weighed in groups of ten to obtain the total biomass per container. The earthworms are placed on the surface of the soil in the container and allowed to burrow (see 11.3). The test containers must be placed into the test chamber randomly.

A1.9.4.1 The worms are purged before weighing if weight loss is to be an endpoint (see 11.6.2.1).

A1.9.5 *Feeding*—It is recommended that food not be added to test containers for tests ≤28 days in duration (see 11.7). Stafford and Edwards **(5)** suggest that the results of a test may be affected by the addition of food due to potential binding properties of the feed and potential selective feeding by the earthworms. In tests longer than 28 days, the use of food may have to be reevaluated, depending on the purpose and endpoints of the test (see A1.9.1.2 and A1.9.1.3).

A1.10 *Biological Data*—Observations may be made at 24 h to evaluate burrowing or non-burrowing without opening the test containers. Mortality and sublethal evaluations may be evaluated on a weekly basis. At the end of the test, the test containers are emptied onto a flat surface, and the earthworms are accounted for and evaluated (see 11.9). Mortality is defined as a lack of response to a gentle mechanical stimulus, for

example, touch with a small spatula or glass rod, to the anterior end of the worm **(37)**. Earthworms may die and decompose within a 14-day testing period, so if all of the individuals are not accounted for at the end of the test, it may be assumed that they died and decomposed completely. Surviving worms may be rinsed with test water and evaluated for behavioral and external pathological endpoints. The following endpoints have been used in various studies: non-burrowing **(6)**, segmental swelling **(6, 47, 77)**, lesions/ulcers **(6, 47, 77)**, coiling **(6, 47, 75)**, shortening/stiffening **(6, 47, 77, 81)**, flaccid/elongated **(6, 77)**, segmental constrictions **(75, 77)**, and tail end autotomy **(82)**. Other endpoints may be developed.

A1.10.1 If weight loss is being used as an endpoint, the surviving earthworms should be washed and purged (see 11.6.2.1) before weighing.

A1.10.2 An *E. fetida* soil toxicity test, independent of duration, is unacceptable if the mean survival of all negative control containers is less than 90 % (see Section 13).

A1.11 *Test Measurements:*

A1.11.1 *pH*—If a concentration series is being tested, the initial pH should be checked in the high and low concentrations at a minimum. If a number of different undiluted site soils are being tested, pH should have already been measured in each soil (see 9.4.3). pH should also be measured in the negative control (and reference soil, if used). Initial pH is measured in a subsample taken from the batch preparation for each treatment.

A1.11.1.1 At the conclusion of a test with a series of concentrations, the pH is checked in subsamples of soil from one of the replicates of the control (and reference soil, if used), high and low concentrations. It is preferable that a replicate without any mortality be used for pH because the process of decay may alter the pH. If a test with undiluted site soils has been terminated, a sample for pH is taken from one replicate of each soil plus the control (and reference soil, if used). Care should be exercised to avoid a sample of soil containing dead worms.

A1.11.2 *Percent Moisture*—If a concentration series is being tested, the initial moisture content may be measured in the high and low concentrations. If a number of different undiluted site soils are being tested, moisture content measurements will have already been measured on the site soils (see 9.4.3). Moisture content may also be measured in the negative control (and reference soil, if used). Initial moisture is measured in subsamples taken from the batch preparation for each treatment and is determined gravimetrically.

A1.11.2.1 At the end of the test, moisture may be measured in one of the replicates of the high and low concentrations and the negative control (and reference soil, if used).

A1.11.3 *Temperature*—A copy of the temperature graph (or temperature/humidity graph) may be attached to the paperwork at the termination of the test (see 11.6.1).

A2. ARTIFICIAL SOIL COMPOSITION

A2.1 The artificial soil (AS) used in this test was developed with the advice of pedologists to overcome the variability between different soil types and has an adsorptive capacity resembling typical loam soils **(37, 107)**. The following constituents are mixed together on a dry weight basis:

(*1*)	Canadian sphagnum (*Sphagnum*) peat moss (that portion passing through a 2.36-mm screen)	10 %
(*2*)	Kaolin clay (97 % kaolinite with a particle size under 40 μm)	20 %
(*3*)	Silica sand (Grade 70, 97.1 % particle size of 0.053 to 0.3 mm)	70 %

A2.1.1 After these materials are mixed together, an amount of calcium carbonate (99 % purity) equal to approximately 0.4 % of their total weight is added to the mixture to adjust the pH to 7.0 ± 0.5. The exact amount of calcium carbonate used will depend on the pH of the peat moss used. For example, 50 kg of AS would have 200 g of calcium carbonate added to it. The materials and source of the materials need to be standardized as much as possible.

A3. BIOACCUMULATION TESTING USING EISENIA FETIDA

A3.1 *Scope:*

A3.1.1 This annex covers the additional procedures required to perform an *Eisenia fetida* bioaccumulation test.

A3.1.2 *Significance—Eisenia fetida* bioaccumulation testing. Bioavailability can not be determined from chemical analysis of the soil alone **(83)**. Earthworm bioassays are an important tool to determine soil toxicity, and potential bioaccumulation with respect to the chemical availability in soil. A method to determine chemical bioavailability and mobility using the earthworm *Eisenia fetida* has successfully evaluated the following chemicals; metals, PAHs, PCBs, pesticides, and butyltins **(2, 9, 84, 86-90, 93)**. The bioaccumulation assay adds information on bioavailability and contaminant mobility of specific chemicals from soil to the soil dwelling earthworms, and the potential for contaminant movement to higher organisms (birds, mammals, fish, amphibians, reptiles, and insects) linked to worms in the food web.

A3.2 *Culture of Test Organisms*—Earthworms are obtained through either culture procedure (see A1.9.1.4) or ordering earthworms. A recent study has shown that reasonable control charts have been maintained with earthworms from an outside source **(85)**.

A3.3 *Age*—Tests with *E. fetida* should use sexually mature

fully clitellate earthworms (see A1.7).

A3.4 *Acclimation*—See 11.5, A1.4, and A1.9.1.4).

A3.5 *Test Specifications:*

A3.5.1 *Experimental Design*—Decisions concerning the various aspects of experimental design, such as the number of replicates, the number of test containers, and the mass of earthworms, should be based on the amount of tissue material needed for chemical analysis.

A3.5.2 *Test Material*—Test materials used have been primarily enriched dredged material. Soils used in this method are the following: soils collected from potentially contaminated sites, reference soils collected from uncontaminated sites, and a negative control material such as earthworm culture media for use in evaluating test acceptability.

A3.5.3 *Test Containers*—Test material is placed in transparent plexiglass cylinders 30 cm deep and 15 cm in diameter. The cylinder ends are closed with a 17-cm in diameter PVC and either 340-μm Nytex mesh or cotton muslin cloth. The bottom end is then placed in a 20-cm diameter plastic dish of test water to allow water movement into the substrate and allow earthworms to move into areas of optimum moisture. (See Fig. A3.1.)

A3.5.4 *Day (0) Test Initiation*—A random sample of earthworms should be analyzed for the chemical(s) of concern as a Day 0 background tissue sample. The Day 0 background tissue sample is used to determine chemicals present in earthworms before the test and should not be confused with any negative control or reference tissue samples which are exposed to test cylinders for the full 28 days and serve to determine test acceptability. If greater than 10 % mortality is seen in a negative control or reference test containers than that test is considered invalid and is rerun. If the test fails a second time it is assumed that the earthworms can not survive in the given soil and therefore contaminant bioaccumulation in the earthworm is not a concern. Prior to testing, earthworms are rinsed with test water, and placed on paper towels to remove excess water. On Day 0 the mass of earthworms needed for the particular chemical analysis procedures for the chemical(s) of concern are added to the test cylinder. Test containers have accommodated up to 30 g (~75 earthworms)/cylinder **(90)**.

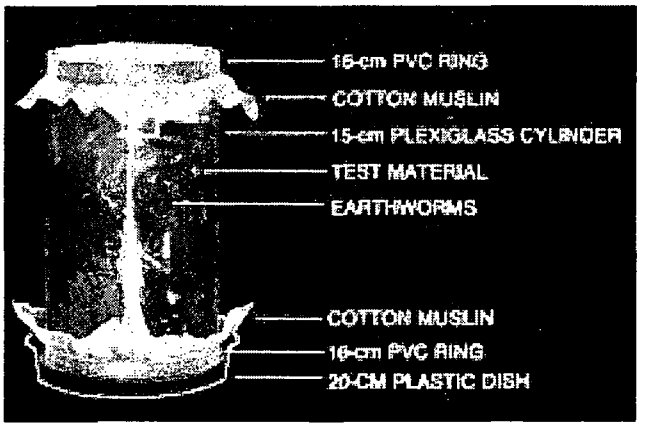

FIG. A3.1 Diagram of the Test Container for the Bioaccumulation Test (see A3.5.3)

A3.5.5 *Day (28) Test Breakdown*—On Day 28, earthworms are removed, rinsed with test water, blotted, counted, and weighed. Depuration of the earthworms is then recommended for 24 h on moist filter paper. Earthworms are then rinsed, reweighed, and frozen in preparation for chemical analysis.

A3.6 *Feeding*—Test materials used have been primarily enriched dredged material, therefore, not requiring an additional food source **(2, 9, 84, 86, 87, 88, 89, 90)**. Soils with less nutrients tested with this procedure may require added food due to test length **(92)**. Any food added would need to be chemically analyzed for concentrations of contaminant(s) of concern. (See A1.9.1.2 and A1.9.1.3)

A3.7 *Quality Control Parameters*—Temperature, pH, percent moisture, and salinity should be controlled and monitored throughout the test. Ideally these parameters should be the same as in the field, and within the range of the earthworms temperature, and pH requirements. Acceptable temperature range is from 10 to 29°C with a recommended range of 19 to 25°C. Acceptable pH range is between 4 and 10 **(17)**. Recommended photoperiod is 24 h within 100 to 1080 lx. This is the same photoperiod suggested for the toxicity test. It is recommended to prevent earthworm escape, encourage maximum exposure to test material, and to discourage contact with container sides. (See Table A3.1.)

A3.8 *Chemical Analysis:*

A3.8.1 *Test Material Analysis*—All test materials should be analyzed for the chemical(s) of concern before test initiation.

A3.8.2 *Tissue Analysis*—A random baseline tissue analysis is performed on Day 0 and all tissues exposed to test cylinders are analyzed on Day 28.

A3.8.3 *Analytical Methodology*—See Section 12.

A3.9 *Test Evaluation*—This bioassay has been used successfully in evaluating contaminant bioavailability and mobility on several projects **(2, 9, 84, 86, 87, 88, 89, 90, 93)**. Data are reported in tables comparing whether the chemical concentrations of Day 28 tissue exposed to the test soil are significantly different from the Day 0 tissue baseline analysis, and the Day 28 tissue exposed to the reference soil. If significantly different, the Day 28 tissue chemical data are discussed as the magnitude above either the Day 0 tissue baseline analysis or the Day 28 tissues from the reference soil (that is, 2×, 5×, 10×). Other approaches for evaluating data include kinetics studies with estimate uptake, depuration rates, and time to steady state, lipid

TABLE A3.1 Test Specifications for the 28-Day *Eisenia fetida* Bioaccumulation Test

Test Duration	28 days
Biological endpoint	contaminant accumulation
Temperature	same as field condition if within 10 to 29°C
Photoperiod	24 h/100 to 1080 lx
pH	same as field condition if within 4 to 10
% moisture	same as field condition
Salinity	same as field condition
Test containers	plexiglass cylinders

normalization and normalizing soil concentrations of non-ionic organics to TOC (see Guide E 1688). Analysis of field collected organisms is also an option.

A3.10 *Test Variations*—Variations on the above procedure have also been successfully used.

A3.10.1 An *in-situ* bioassay using the same procedure as above with a 7.5.1 polyethylene bucket with screen-covered holes in the base and lid to allow air and water but not earthworm exchange. Test containers were implanted 25 cm deep (soil level) and filled with the material removed from the hole **(86)**.

A3.10.2 Another variation was developed with the recommendations to add a more realistic approach to field disposal site conditions by considering effects of natural site vegetation **(92)**. This variation is conducted with Bermuda grass planted in the cylinders **(90)**. The procedure differs as follows; On day 0, 1 gm of Bermuda grass seeds are spread over the cylinder surface. Seeds are covered with 1 mm of peat moss and lightly watered with RO water. Each cylinder received 125 mL of a dilute (600 mg/L of water) solution of soluble plant food (13-13-13), during the first two weeks to enhance seed sprouting. Excess water collecting in plastic trays was poured off. On Day 30 earthworms are added. On Day 60 Bermuda grass is harvested, earthworms are counted, weighed, and both are prepared for chemical analysis. The following alterations are made in the temperature and lighting test conditions to promote grass growth: temperature 22°C (night) to 29°C (day), acceptable lighting for this study is 400 lux illumination for a period of 14 h light/10 h dark.

A4. ENCHYTRAEIDAE REPRODUCTION TEST

A4.1 *Scope*:

A4.1.1 This standard annex of Guide E 1676 covers the additional or modified procedures required to perform an Enchytraeid Reproduction Test (ERT) from Guide E 1676 for Conducting a Laboratory Soil Toxicity or Bioaccumulation Test with the Lumbricid Earthworm *Eisenia fetida*.

A4.2 *Significance—Enchytraeus albidus* (Henle, 1837), Oligochaeta, has been selected (together with other species of the genus *Enchytraeus*) as a test species for the following reasons **(104-106, 118)**: *(1)* it has a short generation time; *(2)* it reproduces very well in the laboratory; *(3)* it can easily be kept and cultured in the laboratory; *(4)* data on it's survival, growth, and reproduction are available from the literature; *(5)* it is a representative of an ecologically relevant family of soil organisms, especially in acidic soils **(94)**. *E. albidus* seems to be sensitive towards different anthropogenic stress factors like pesticides or heavy metals **(97-100)**. It has been used successfully as a laboratory test organism in many testing media, for example, artificial soil **(104)**, contaminated field soils **(109)**, sediment **(110)**, agar **(105)**, and water **(111)**. Basic information on the ecology and ecotoxicology of enchytraeids in the terrestrial environment can be found in Refs **(94, 95, 96, 104, 112, 113, 114)**.

A4.3 *Life History*—Like *E. fetida*, the life cycle of *E. albidus* and other species of this genus can be divided into three phases: *(1)* the cocoon phase, *(2)* the juvenile (immature) phase, *(3)* and the adult (mature) phase (see A1.2). Its life cycle is short as maturity is reached between 33 days (at 18°C) and 74 days (at 12°C); that is, from freshly deposited cocoon through clitellate worm and deposition of the next generation of cocoons **(104, 105)**. In the case of *E. albidus*, reproduction is strongly inhibited at temperatures higher than 22 to 25°C, whereas other, mainly smaller, species of the same genus produce cocoons at temperatures between 25 and 30°C **(117)**. Despite the fact that *E. albidus* individuals have been kept under optimal laboratory conditions for more than 1.5 years, an age of less than one year is more usual in the field. The length of an adult *E. albidus* is usually 15 mm, but can vary, depending on nutrition, between 10 and 35 mm.

A4.3.1 *E. albidus* is found in marine, limnic, and terrestrial habitats worldwide, mainly in decaying organic matter (seaweed, compost) and rarely in meadows **(95, 115)**. The worms can be kept for up to four days in water **(111)**. In general, many species of the genus *Enchytraeus* are known to be among the first enchytraeids colonizing new biotopes or belong to the dominant species at disturbed sites (for example, urban soils) **(115)**, while others can be found in all other terrestrial habitats as well as in limnic and marine sediments **(95, 101, 102)**.

A4.3.2 The rate of organic matter consumption in the laboratory for *E. albidus* is not known. The specific sources of nutrition for this species are not well understood. It is known that the worms can take up amino acids directly from the surrounding aquatic phase **(108)**, that they feed on microorganisms (especially bacteria) from decaying organic material (including dead earthworms), and that they are even able to divide leaves and digest this nearly intact plant material **(116)**. Often mineral debris is taken up along with the organic material.

A4.4 *Taxonomy*—The test species *E. albidus* belongs to the genus *Enchytraeus* sp. (order Oligochaeta, class Clitellata, phylum Annelida). Henle (1837) scientifically described it as the first member of the new family Enchytraeidae. In the meantime, approximately 116 species have been described in the genus *Enchytraeus* sp. worldwide, but many of these descriptions are not valid. The taxonomic status of nearly all *Enchytraeus* species has to be revised. Species determination is only possible morphologically, if at all, with adult animals since juveniles do not have sexual organs like sperm ducts. *E. albidus* is not only the type species for the whole family but also the best known species, which has been used in ecotoxicology, physiology, biochemistry, and genetics for more than 50 years.

A4.4.1 Some morphological features of *E. albidus* can be quite variable, especially the spermatheca. So, considering its wide geographical and ecological range, it has been proposed that it is actually not one but a group of closely related species.

However, no evidence of this has been found up to now, e.g. by means of biochemical or genetic methods. *E. albidus* shows some morphological features which are easy to detect even for those not experienced in enchytraeid taxonomy. This species can be distinguished quite easily from all other species in the genus *Enchytraeus*: *(1)* it is the largest species of this genus (except some subantarctic species), *(2)* it is the only terrestrial species having four setae per bundle in at least some segments (usually in the head region), and *(3)* it has a very unique and quite and has an obviously long seminal duct, which extends through the clitellum region and several segments beyond **(103, 104)**.

A4.4.2 When in cultures, because of slight differences in their ecological demands, *E. albidus* is outnumbered by other, usually smaller and faster reproducing species of this genus. Such animals can only determined by specialists, since very often not only morphological but also enzymatic parameters are necessary. Therefore, when another species has to be selected for testing purposes, only worms from a well-defined source should be used for this purpose. Some helpful guidance on species determination can be found in Nielsen and Christensen **(102)** and Bougouenec and Giani **(117)**.

A4.5 *Culture of Test Organisms*—Since the beginning of this century, at least, *E. albidus* was bred as fish food to be used in aquaria **(118)**. Even cultures on a "field scale" were recently considered in Canada, Russia, and France (for example, **119**).

A4.5.1 *E. albidus* (as well as other *Enchytraeus* species) can be bred in large plastic boxes (for example, 30 by 60 by 10 cm) filled with a mixture of artificial soil and natural, uncontaminated garden soil. Compost material should be avoided since it could contain toxic substances like heavy metals. Fauna should be removed from the breeding soil before use. Pure artificial soil can also be used but the reproduction rate could be slower compared to that obtained with mixed substrates. The substrate should have a pH of 6.0 ± 0.5.

A4.5.2 The culture should be kept in an incubator at a temperature of 15 ± 2°C without light. A temperature higher than 23°C should be avoided. The artificial/natural soil moisture should be moist but not wet. When the soil is gently pressed by hand, only small drops of water should appear. In any case, anoxic conditions should be avoided (e.g. if a lid is used, the number of lid holes should be high enough). The breeding soil can be aerated by carefully mixing it once per week.

A4.5.3 The worms can be fed approximately twice a week with a proper amount of oatmeal flakes (rolled oats) which are strewn on the soil surface or carefully mixed into the substrate at least every two weeks. If food from the last feeding date remains on the soil surface, the amount of food given should be adjusted accordingly. If fungi grow on the remaining food, it should be replaced by a new quantity of rolled oats. From time to time, the rolled oats can be supplemented with commercially purchased vitamins, milk and cod liver oil. After three months, the animals can be transferred into a freshly prepared culture or breeding substrate. No carrying capacities have been calculated so far.

A4.5.4 The rolled oats, which should be stored in sealed vessels, can be autoclaved or heated before use to avoid infections by flour mites (for example, *Glyzyphagus* sp., Astigmata, Acarina) or predaceous mites (for example, *Hypoaspis* (*Cosmolaelaps*) *miles*, Gamasida, Acarina). None of these animals in low numbers appears to be a problem for healthy worms. After this procedure, the food can be ground up so that it can easily be strewn on the soil surface. Another possible food source is baker's yeast or the fish food "Tetramin."

A4.5.5 In general, the culturing conditions are sufficient if worms *(a)* do not try to leave the substrate, *(b)* move quickly through the soil, *(c)* exhibit a shiny outer surface without soil particles clinging to it, *(d)* are more or less whitish colored, and *(e)* if worms of different ages are visible. Generally, worms can be considered to be healthy if they reproduce continuously.

A4.6 *Obtaining Brood Stock—E. albidus* starter cultures can be obtained from *(1)* laboratories or universities working in soil ecology and *(2)* local aquarium stores. In the latter case, an expert should confirm species determination.

A4.7 *Age*—The animals used in the tests should be adult worms. They should have eggs (white spots) in the clitellum region, and they should have approximately the same size (≈1 cm). Synchronization of the breeding culture is not necessary.

A4.8 *Handling—E. albidus* should be handled as little as possible. When handling is necessary, it should be done as gently, carefully, and quickly as possible, so that the worms are not stressed unnecessarily. Any worms that are dropped or injured during handling should be discarded.

A4.9 *Selection and Acclimatization*—Before testing, the enchytraeids should be acclimated to the soil used for the tests under the test conditions (including feeding) for at least 24 h. A higher number of adult worms than that needed for performing the test is used. At the end of the acclimation period, only worms with eggs and showing no behavioral anomalies (for example, trying to escape from the soil) should be selected for the test. The selected worms are placed in a petri dish filled with a small amount of water to be observed with a stereomicroscope and the animals that have no eggs are discarded. Freshwater is preferred to demineralized water or tap water (possible copper contamination) which could be harmful to the enchytraeids. The other organisms living in the cultures such as mites, should also be removed.

A4.10 *Toxicity Test Specifications*:

A4.10.1 *Introduction*—This test is designed to assess the effects of chemicals on the reproductive output of the enchytraeid worm (*Enchytraeus albidus*). It is based principally on a method developed by the Umweltbundesamt, Germany **(104)**. Other methods for testing the toxicity of chemicals to Enchytraeidae and other earthworms have also been considered **(105, 106)**. Adult enchytraeid worms are exposed to a range of concentrations of the test substance mixed in an artificial soil. The test can be divided into two steps: *(a)* a range-finding test in which mortality is the main endpoint assessed after two weeks exposure and *(b)* a definitive reproduction test in which the total number of juveniles produced by parent animals and the survival of parent animals

are assessed. The test duration is six weeks. After the first three weeks the adult worms are removed and morphological changes (for example,. open wounds) are recorded. After an additional three weeks, the number of offspring, hatched from the cocoons, is counted. The reproductive output of the animals exposed to the test substance is compared to that of the control(s) to determine the no observed effect concentration (NOEC). As far as possible, the data are also analyzed using a regression model to estimate the concentration that would cause a x % reduction in reproductive output, that is, ECx (for example, EC_{10}, EC_{50}).

A4.10.2 *Design for the Range-Finding-Test*—When necessary, a range-finding test should be conducted with five concentrations of the test substance. One replicate for each treatment and the control is desirable. The main endpoint is mortality.

A4.10.2.1 The test duration is two weeks. At the end of the test, mortality of the worms should be assessed by carefully searching the substrate for surviving individuals (for example, using a spatula). An animal is recorded as dead if it does not respond to a gentle mechanical stimulus to the front end. Moreover, changes in behavior (for example, inability to dig into the soil; lying motionless against the glass wall of the test vessel) and in morphology (for example, open wounds), should be recorded. Likewise, the presence of juveniles can be observed by using the staining method (see A4.10.9). This will help select the test concentrations for the definitive test.

A4.10.2.2 Probit analysis **(121)** should be applied to determine the LC_{50}. In case of failure (for example, if data from less than three concentrations with partial kills are available), alternative methods can be used such as moving averages **(122)** or simple interpolation (for example, geometrical mean of LC_0 and LC_{100}, as computed by the square root of LC_0 multiplicated by LC_{100}).

A4.10.2.3 The LC_{50} should be used to determine the concentration range for the definitive test. The NOEC or the EC_{10} for reproduction are assumed to be lower than the LC_{50} by a factor up to ten. However, this is an empirical relationship and it might be different in a given case. Therefore, additional endpoints or observations or both in the range-finding test, such as the occurrence of juveniles, can help refine the test concentration range to be used for the definitive test.

A4.10.2.4 If a more accurate determination of the LC_{50} is required, the test should be performed using eight concentrations of the test substance, with four replicates for each test concentration and eight replicates for the controls.

A4.10.3 *Definitive Reproduction Test*—The endpoint is fecundity (for example, the number of juveniles produced). As in the range-finding test, all other harmful signs should be recorded. Three options for the design for the definitive reproduction test are described in 14.7.

A4.10.3.1 Ten adult worms per test vessel should be used. The animals are fed at the beginning of the test and then once a week. After 21 days, living adult worms are counted and changes in behavior (for example,. inability to dig into the soil; lying motionless against the glass wall of the test vessel) and in morphology (for example, open wounds) should also be recorded. Then, all adult worms are removed as in A4.10.2.1. The test soil (that is, without the parent worms and containing the cocoons laid down) is incubated for three additional weeks under the same test conditions, including food supply until Day-28.

A4.10.3.2 After six weeks, the newly hatched worms are isolated and counted using Bengalred staining (see A4.10.9; 120). Wet (but not heat) isolation techniques have proved to be suitable **(104, 107, 113)** and may also be used. However, the method using Bengalred is preferred since the wet isolation from a soil substrate is hampered by the clay particles that make the water turbid.

A4.10.3.3 If no effects are observed at the highest concentration in the range-finding test (that is, 1000 mg/kg), the reproduction test can be performed as a limit test, using 1000 mg/kg to demonstrate that the NOEC or the EC_{10} for reproduction is greater than this value. The number of replicates should be eight for both the test concentration and control.

A4.10.4 *Equipment*—The test vessels should be made of glass or other chemically inert material. The test vessels are glass jars with glass lids (volume: 0.20 to 0.25 L; diameter: ≈6 cm). The lids allow for air exchange and they also reduce water evaporation. Normal laboratory equipment and especially the following should be used: drying cabinet; stereomicroscope; pH and lux meters; suitable accurate balances; adequate equipment for temperature control; adequate equipment for humidity control; incubator or small room with air conditioner; jewelers tweezers, hooks, or loops; and photo basins with ribbed bottoms.

A4.10.5 *Test Substrate*—Other potential test substrates are *(1)* reference soils or potentially toxic site soils; *(2)* artificial, reference, or site soils spiked with compounds; *(3)* site soils diluted with reference soils; or *(4)* site or reference soils diluted with artificial soil.

A4.10.5.1 The composition of artificial soil is described in detail in Annex A2 **(107)**. The dry constituents of the soil are mixed thoroughly (for example, in a large-scale laboratory mixer). This should be done about one week before starting the test. The mixed soil should be stored for at least two days to equilibrate/stabilize the acidity. For the determination of pH, a mixture of soil and 1M KCl solution in a 1:5 ration is used. If the pH value is not within the required range (6.0 ± 0.5), a sufficient amount of $CaCO_3$ is added or a new batch of soil is prepared.

A4.10.5.2 The maximum water-holding capacity (WHC) of the artificial soil should be determined. One or two days before starting the test, the dry artificial soil is moistened by adding enough deionized water to obtain approximately half of the final water content, that is, 40 to 60 % of the maximum WHC (corresponding to 50 ± 10 % moisture dry mass). At the start of the test, the premoistened soil should be divided into as many batches as the number of test concentrations and controls used for the test, and the moisture content should be adjusted to 40 to 60 % by using the solution of the test substance or by adding distilled or deionized water or both. The moisture content should be determined at the beginning and at the end of the test (at 105°C). It is be optimal for the worms' life (the moisture can also be checked as follows: when the soil is

gently squeezed in the hand, small drops of water should appear between the fingers).

A4.10.5.3 *Effect of Grain Size, Organic Carbon, and Moisture on the Test Organisms*—The potential effects of these soil properties on test organisms are not known. This limitation is especially important when using field-collected soils for which no reference control soil (that is, an uncontaminated soil having the same properties as the test soil) is used.

A4.10.6 *Test Groups and Controls*—For each test concentration, an amount of test soil corresponding to 20-g dry weight should be placed into the test vessel. Controls, without the test substance, are also prepared. Food is added according to A4.10.8. In each test vessel, ten worms should be placed carefully on the soil surface (for example, using jeweler's tweezers, hooks, or loops). The collected worms are randomly allocated to test vessels. The number of replicates for test concentrations and for controls depends on the test design used. All test vessels should be randomly placed in the incubator and they should be moved every week.

A4.10.6.1 If a solvent is used for application of the test substance, one control series containing the solvent should be run in addition to the test series. The solvent or dispersant concentration should be the same as that used in the test vessels containing the test substance (see 9.5.3.4). Alternativly, only the highest solvent concentration can be tested.

A4.10.7 *Test Conditions*—The test temperature should be 20 ± 2°C. To avoid worms escaping from the soil, the tests are carried out under controlled light-dark cycle of long-day conditions (preferably 16 to 8 h at 400 to 800 lux in the area of the test vessels).

A4.10.7.1 The vessels should be covered with glass lids which help reduce water evaporation. To check the soil humidity, the vessels should be weighed at the beginning of the test and furthermore once a week, and the weight loss should be replenished with the appropriate amount of deionized water. Loss of water can also be diminished by keeping a high air humidity (>80 %) in the test incubator.

A4.10.7.2 The moisture content and the pH should be measured at the beginning and the end of both the range-finding test and the definitive test. This should be done using an additional sample of the test soil containing no worms. The same amount of food as in the other vessels should be added to these additional vessels at the beginning of the test; indeed, the measured parameters may be influenced by the soil microbial activity. It is not necessary to add food to these vessels during the test.

A4.10.8 *Feeding*—Any food capable of maintaining the enchytraeid population can be used. Commercially purchased rolled oats, preferably autoclaved before use to avoid microbial contamination (heating is also appropriate), were found to be suitable. For each test vessel, the first feeding should be made by mixing 50 mg of ground rolled oats with the soil containing the test substance before placing the worms. Afterwards, weekly food supplies, consisting of 25 mg of ground rolled oats per vessel, should be given, except after 28 days (feeding is not necessary since the juveniles are too small), by putting the food on the surface of the soil taking care not to injure the worms. To reduce fungal growth, the oats flakes should be sunk into the soil (for example, small pieces of soil can be moved to the top of the oat flakes). The flakes should not be completely incorporated, since this procedure might harm the worms. In case the worms do not consume the whole food provided, food supply should be reduced accordingly to avoid fungal growth or molding.

A4.10.9 *Isolating Techniques for Juvenile Worms*:

A4.10.9.1 *Staining with Bengalred*—This method, originally developed in limnic ecology, was first proposed for the counting of juvenile enchytraeids in the enchytraedae reproduction test by W. de Coen **(120)** Independently, a modified version (Bengalred mixed with formaldehyde instead of ethanol) was developed by RIVM Bilthoven **(106)**. At the end of the definitive test (thati is, after six weeks), the artificial soil in the test vessels should be transferred to a shallow container (for example, a Bellaplast vessel or to a photo basin with ribbed bottom) and the juveniles are fixed with ethanol (approximately 5 mL per replicate/vessel). Then the vessels should be filled with water up to a layer of 1 to 2 cm. Afterwards, a few drops (200 to 300 mL) of Bengalred (1 % solution in ethanol) should be added (0.5 % eosin might be an alternative) and the two components are mixed carefully. After 12 h, the worms are completely reddish colored. Now it is very easy to count them because they are lying on the surface of the substrate. Another possibility is to press the substrate/alcohol mixture through a sieve (mesh size: 0.250 mm) before counting the worms. The kaolinite, the peat, and some sand grains are lost and the reddish colored worms are easier to see. The use of illuminated lenses (lens size at least 100 by 75 mm; magnification factor 2 to 3×) also facilitates counting the already reddish juveniles. Thanks to this improvement, the counting time is reduced to a few minutes per vessel. Using the staining method, the vessels of one test can be assessed by a single person within one day (maximum two days) some hours or days after the end of the test.

A4.10.9.2 *Wet Removal of Juvenile Worms* **(112, 113)**—The removal of juvenile worms should be started immediately after the end of the test. The artificial soil of each test vessel should be placed into a common plastic or stainless steel sieve. The sieves are put in plastic bowls without touching the bottom. The bowls are carefully filled up with water until the samples in the sieves are completely under the water surface. To ensure a recovery rate of more than 90 %, the removal should occur within three days at 20 ± 2°C (that is, the worms have enough time to move from the soil through the sieve into the water). Once the worms are isolated, the sieves are removed and the water (except for a small amount) is slowly decanted. The sediment at the bottom of the bowls should not be disturbed. Then the plastic bowls are shaken slightly to suspend the soil in the overlying water, which is transferred to a petri dish. After clarification of the water (that is, the soil particles have settled), the enchytraeids can now be collected out of the petri dish under a stereomicroscope using a softsteel forceps.

A4.10.9.3 *Flotation*—Alternatively according to a note by R. Kuperman (U.S. Army), the following procedure is also possible **(128)**: After fixing the content of a test vessel with ethanol, the artificial soil is flooded with Ludox (AM-30 colloidal silica, 30 wt. % suspension in water) up to 10 to 15

mm above the soil surface. After thoroughly mixing the soil with the flotation agent, the juvenile worms floating on the surface can easily be counted after 2 to 3 min.

A4.10.10 *Test Acceptability Requirements*—For the test to be valid, the following performance criteria must be met in the controls: *(1)* the mortality does not exceed 20 % at the end of the range-finding test and after the first three weeks of the reproduction test, *(2)* the average number of juveniles is higher than 25 per test vessel at the end of the test, assuming that 10 adult worms per test vessel were used, and *(3)* the coefficient of variation around the mean number of juveniles is not higher than 50 % at the end of the reproduction test.

A4.10.11 *Reference Substance*—A reference substance should be tested once a year or possibly included in the test series. A suitable reference substance is carbendazim, which has been shown to affect survival and reproduction of enchytraeids **(100)**. The EC_{50} for reproduction should be in the range of 1.2 ± 0.8 mg a.i./kg dry mass **(104)**. If a positive toxic standard is included in the test series, one concentration is used and the number of replicates should be the same as that in the controls, that is, eight replicates. For carbendazim, the testing of 1.2 mg a.i./kg dry weight (tested as liquid formulation) is recommended.

A4.10.12 *Performance with Other Enchytraeus Species than E. albidus*:

A4.10.12.1 *Selection of Species*—Species other than *E. albidus* may be used but the test procedure and the validity criteria should be adapted to provide suitable test conditions. Many *Enchytraeus* species are readily available and can be satisfactorily maintained in the laboratory. Therefore, the most important criterion for selecting an *Enchytraeus* species other than *E. albidus* is ecological relevance and, additionally, comparable sensitivity. There may also be formal reasons for a change of species. In countries in which *E. albidus* does not occur and cannot be imported (for example, because of quarantine restrictions), other *Enchytraeus* species may be used. Potential candidates are listed in the following.

A4.10.12.2 *Enchytraes crypticus (Westheide & Graefe 1992)*—In recent years, this species has often been used in ecotoxicological studies because of the simplicity of its breeding and testing **(106, 109)**. However, its individual size is small, which makes handling more difficult than with *E. albidus* (especially before implementation of the staining method). Additionally, it was only described from earthworm cultures. Since this species has not been found to exist with certainty in the field up to now, its ecological requirements are not known.

A4.10.12.3 *Enchytraeus buchholzi (Vejdovsky 1879)*—This name probably covers a group of closely related species which are morphologically difficult to distinguish. Therefore, its use is not recommended until the animals used in a test are clearly described. From an ecological standpoint, these animals are usually found in meadows and disturbed sites like roadsides.

A4.10.12.4 *Enchytraeus luxurious (Schmelz and Collado, 1999)*—U. Graefe (Hamburg) found this species for the first time in a meadow close to St. Peter-Ording (Schleswig-Holstein, Germany). Because of its size, it could be a good alternative to *E. albidus*.

A4.10.12.5 *Enchytraeus bulbosus (Nielsen and Christensen 1963)*—This species has hitherto been reported from German and Spanish mineral soils, where it is common but usually not very abundant. In comparison to other small species of this genus, it is relatively easy to determine. Additionally, *E. bulbosus* seems to be easy to culture (E. Belotti, personal communication). Up to now, however, nothing is known about its behavior in laboratory tests and about its sensitivity to chemicals.

A4.10.12.6 *Breeding Conditions*—All *Enchytraeus* species mentioned previously can be kept and bred in the same substrate as *E. albidus*. The size of the breeding vessels can be smaller. They can also be fed the same food (that is, rolled oats), but because of their smaller individual size, the amount of food per feeding should be adjusted. In general, it should be kept in mind that the lifecycle of these animals is shorter, which means, for example, that feeding should be done more often.

A4.10.12.7 *Test Conditions*—The conditions are the same as in the case of *E. albidus*, except for the following aspects: *(1)* the size of the test vessel may be smaller; *(2)* the duration of the reproduction test may be shorter, that is, four instead of six weeks; the duration of the Range-finding test should not be changed; *(3)* because of the small individual size of the juvenile worms the use of the staining method is strongly recommended for counting; and *(4)* the value for the validity criterion "number of juveniles per test vessel in the control" should be changed to "50."

A4.10.13 *Treatment of Results*—See Section 14.

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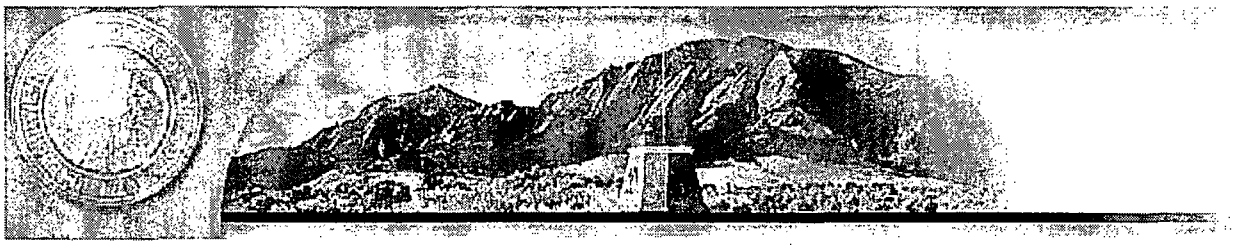

The In-Vitro Method

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University of Colorado

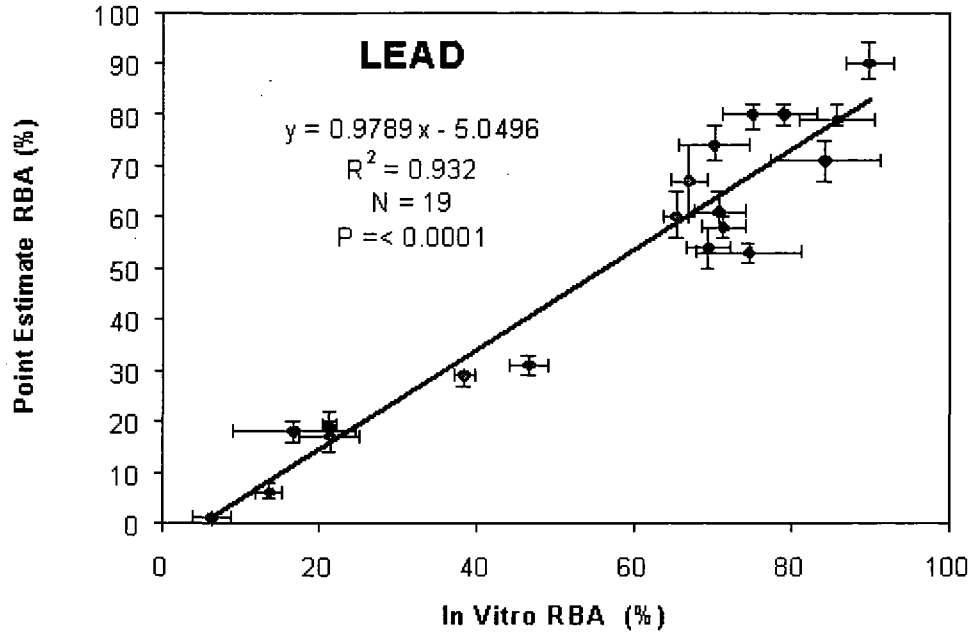
Relative Bioavailability Leaching Procedure

Standard Operating Procedure

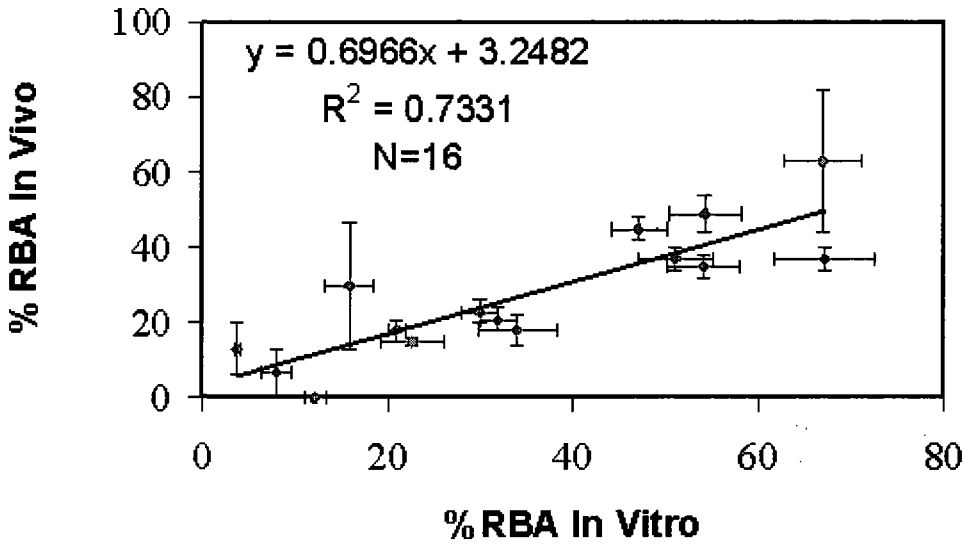
1.0 Purpose

An increasingly important property of contaminated media found at environmental sites is the bioavailabilty of individual contaminants. Bioavailability is the fraction of a contaminant that is absorbed by an organism via a specific exposure route. Many animal studies have been conducted to experimentally determine oral bioavailability of individual metals, particularly lead and arsenic. During the period 1989-97, a juvenile swine model developed by USEPA Region VIII was used to predict the relative bioavailability of lead and arsenic in approximately 20 substrates (Weis and LaVelle 1991; Weis et al. 1994). The bioavailability determined was relative to that of a soluble salt (i.e. lead acetate trihydrate or sodium arsenate). The tested media had a wide range of mineralogy, and produced a range of lead and arsenic bioavailabilty values. In addition to the swine studies, other animal models (e.g. rats and monkeys) have been used for measuring the bioavailabilty of lead and arsenic from soils.

Several researchers have developed in vitro tests to measure the fraction of a chemical solubilized from a soil sample under simulated gastrointestinal conditions. The in vitro tests consist of an aqueous fluid, into which the contaminant is introduced. The solution than solubilizes the media under simulated gastric conditions. Once this procedure is complete, the solution is analyzed for lead and/or arsenic concentrations. The mass of the lead and/or arsenic found in the filtered extract is compared to the mass introduced into the test. The fraction liberated into the aqueous phase is defined as the bioavailable fraction of lead or arsenic in that media. To date, for lead-bearing materials tested in the USEPA swine studies, this in vitro assay has correlated well ($R2 = 0.93$, $p = .0001$) with relative bioavailability. Arsenic has yet to be fully validated but shows a promising correlation with in vivo results.



ARSENIC



by EMPA techniques and for which bioavailability results from acceptable animal stu available have been used for this study. A total of 20 substrates have been tested in va relative bioavailability leaching procedure (RBLP).

3.0 Relevant Literature

Background on the development and validation of in vitro test systems for estimating

arsenic bioaccessibility can be found in; Ruby et al. (1993, 1996); Medlin (1972); Medlin and Drexler, 1997; Drexler, 1998; and Drexler et al., 2003.

Background information for the USEPA swine studies may be found in (Weis and LaVelle, 1991; Weis et al. 1994; and Casteel et al., 1997) and in the USEPA Region VIII Center in Denver, Colorado.

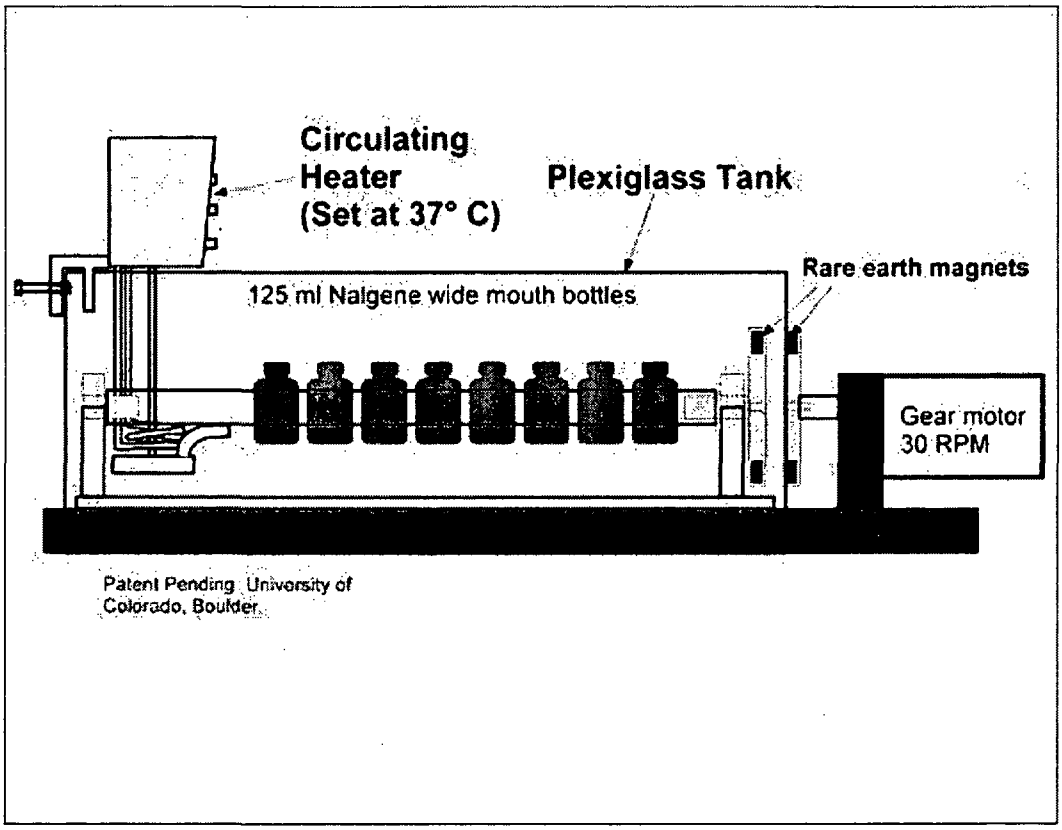
4.0 Sample Preparation

All media are prepared for the in vitro assay by first drying (<40 °C) all samples and then sieving to < 250 m m. The <250 micron size fraction was used because this is the particle size is representative of that which adheres to children's hands. Samples were thoroughly mixed prior to use to ensure homogenization. Samples are archived after the study completion and retained for further analysis for a period of six months unless otherwise requested. Prior to obtaining a subsample for testing in this procedure, each sample must be homogenized in its sample container by end-over-end mixing.

5.0 Apparatus and Materials

5.1 Equipment

The main piece of equipment required for this procedure is the extraction device illustrated in Figure 1. The device can be purchased from the Department of Geological Sciences, University of Colorado. For further information contact Dr. John W. Drexler, at (303) 492-5251 or drexlerj@spot.colorado.edu. The device holds ten 125 ml, wide-mouth high-density polyethylene (HDPE) bottles. These are rotated within a Plexiglas tank by a TCLP extractor motor with a modified flywheel. The water bath must be filled such that the extraction bottles remained immersed. Temperature in the water bath is maintained at 37 +/- 2 °C using an immersion circulator heater (Fisher Scientific Model 730).



The 125-ml HDPE bottles must have an airtight screw-cap seal (Fisher Scientific #02-893-5C), and care must be taken to ensure that the bottles do not leak during the extraction procedure.

5.2 Standards and Reagents

The leaching procedure for this method uses an aqueous extraction fluid at a pH value of 1.5. The pH 1.5 fluid is prepared as follows:

Prepare 2 L of aqueous extraction fluid using ASTM Type II demonized (DI) water. The buffer is made up in the following manner. To 1.9 L of DI water, add 60.06 g glycine (free base, reagent grade), and bring the solution volume to 2 L (0.4M glycine). Place the mixture in the water bath at 37 °C until the extraction fluid reaches 37 °C. Standardize the pH meter (one should use both a 2.0 and a 4.0 pH buffer for standardization) using temperature compensation at 37 °C or buffers maintained at 37 °C in the water bath. Add trace metal grade, concentrated hydrochloric acid (12.1N) until the solution pH reaches a value of 1.50 +/_ 0.05 (approximately 60 mL).

All reagents must be free of lead and arsenic, and the final fluid must be tested to confirm that lead and arsenic concentrations are less than one-fourth the project required detection limits (PRDLs) of 10 and 20 µg/L, respectively (e.g., less than 2 µg/L lead and 5µg/L arsenic in the final fluid.

Cleanliness of all materials used to prepare and/or store the extraction fluid and

buffer is essential. All glassware and equipment used to prepare standards and reagents must be properly cleaned, acid washed, and finally, triple-rinsed with demonized water prior to use.

6.0 Leaching Procedure

Add 1.00 +/- 0.5 g of test substrate (<250m m) to the bottle, ensuring that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle. If necessary, use an antistatic brush to eliminate static electricity prior to adding the media. Record the mass of substrate. When ready to begin the test--measure 100 +/- 0.5 mL of the extraction fluid, using a graduated cylinder or auto pipette and transfer to the 125 mL wide-mouth HPDE bottles. Hand-tighten each bottle top and shake/invert to ensure that no leakage occurs, and that no media is caked on the bottom of the bottle.

Place the bottle into the modified TCLP extractor, making sure each bottle is secure and the lid(s) are tightly fastened. Fill the extractor with 125 mL bottles containing test materials or QA samples.

The temperature of the water bath must be 37 +/- 2 °C.

Turn on the extractor and rotate end-over-end at 30 +/- 2 rpm for 1 hour. Record the start time of rotation.

When extraction (rotation) is complete, immediately stop the extractor rotation and remove the bottles. Wipe them dry and place upright on the bench top.

Draw extract directly from the reaction vessel into a disposable 20 cc syringe with a Luer-Lok attachment. Attach a 0.45 µm cellulose acetate disk filter (25 mm diameter) to the syringe, and filter the extract into a clean 15 mL polypropylene centrifuge tube (labeled with sample ID) or other appropriate sample vial for analysis.

Record the time that the extract is filtered (i.e. extraction is stopped). If the total time elapsed is greater than 1 hour 30 minutes, the test must be repeated.

Measure the pH of the remaining fluid in the extraction bottle. If the fluid pH is not within +/_ 0.5 pH units of the starting pH, the test must be discarded and the sample reanalyzed as follows:

If the pH has changed more than 0.5 units, the test will be re-run in an identical fashion. If the second test also results in a decrease in pH of greater than 0.5 s.u. this will be recorded, and the extract filtered for analysis. If the pH has increased by 0.5 s.u. or more, the test must be repeated, but the extractor must be stopped at specific intervals and the pH manually adjusted down to pH of 1.5 with dropwise addition of HCl (adjustments at 5, 10, 15, and 30 minutes into the extraction, and upon final removal from the water bath { 60 min}). Samples with rising pH values might better be run following the method of Medlin, 1997.

Store filtered samples in a refrigerator at 4 °C until the are analyzed. Analysis for lead and arsenic concentrations must occur within 1 week of extraction for each sample.

Extracts are to be analyzed for lead and arsenic, as specified in EPA methods 6010B, 6020, or 7061A.

6.1 Quality Control/Quality Assurance

Quality Assurance for the extraction procedure will consist of the following quality control samples.

Bottle Blank-extraction fluid only run through the complete procedure at a frequency of 1 in 20 samples.

Duplicate sample-duplicate sample extractions to be performed on 1 in 10 samples.

Matrix Spike-a subsample of each material used will be spiked at concentrations of 10 mg/L lead and 1 mg/L arsenic and run through the extraction procedure (frequency of 1 in 10 samples).

National Institute of Standards and Testing (NIST) Standard Reference Material (SRM) 2711 will be used as a control soil. The SRM will be analyzed at a frequency of 1 in 20 samples.

Control limits and corrective actions are listed in Table 1.

	Analysis Frequency	Control Limits
Bottle blank	5% - 1:20	< 25 µ g/L lead
Blank spike *	5% - 1:20	85-115% recovery
Matrix spike *	10% - 1:10	75-125% recovery
Duplicate sample	10% - 1:10	+/- 20% RPD**
Control soil ***	5% - 1:20	+/- 10% RPD

* Spikes contained 10 mg/L lead and arsenic.

** RPD= relative percent difference.

*** The National Institute of Standards and Technology (NIST) Standard Reference Material (SRM)

7.0 Chain-of-Custody Procedures

All media once received by the Laboratory must be maintained under standard chain-of-custody.

8.0 Data Handling and Verification

All sample weights, fluid concentrations ,and calculations must be recorded on data sheets. Finally all key data will be entered into the attached EXCEL spreadsheet for final delivery and calculation of relative bioavailability.

9.0 References

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SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

STATIC WATER LEVEL, TOTAL WELL DEPTH, AND IMMISCIBLE LAYER MEASUREMENT

SOP NO. 014

REVISION NO. 0

Last Reviewed: December 1999

Quality Assurance Approved

July 20, 1994

Date

1.0 BACKGROUND

Measurement of static water level, total well depth, and any immiscible layers is necessary before a well can be sampled and groundwater flow direction can be determined. If an immiscible layer is present, its depth and thickness must be determined. In addition, the static water level and total depth of a monitoring well are needed to determine a purging volume.

1.1 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide guidelines for field personnel measuring static water levels and total water depths of monitoring wells or piezometers. This SOP also provides guidelines for measuring immiscible layers in such wells.

1.2 SCOPE

This SOP describes the methodologies for measuring static water level, total well depth, and immiscible layer depth and thickness.

1.3 DEFINITIONS

To clarify the methodologies presented in this SOP, the following definitions are presented:

Electrical Water Level Indicator: An electrical probe used to determine the depth to fluid. The probe has a light or sound alarm connected to an open circuit. The circuit is closed and the alarm is activated when the probe contacts a conducting fluid such as water.

Immiscible Layer: A liquid phase that cannot be uniformly mixed or blended with water. Heavy immiscible phases sink in water; light immiscible phases float on water.

Interface Probe: An electrical probe used to determine the thicknesses of light or dense immiscible layers in the water column of a monitoring well.

Ionization Detector: A photoionization detector (PID) or a flame ionization detector (FID) is used to measure the level of volatile organic compounds in the gaseous phase. These units are generally not compound-specific and thus measure only total volatile organic compounds. The PID generally cannot detect as complete a range of compounds as the FID. This difference is the result of the relative ionization energies of the two detectors. Most PIDs cannot detect methane, but FIDs can. The HNu and Microtip are examples of PIDs; the Foxboro organic vapor analyzer (OVA) is an example of an FID.

Static Water Level: The level of water in a monitoring well or piezometer. This level can be measured as the depth to water or as the elevation of water relative to a reference mark or datum.

Total Well Depth: The distance from the ground surface to the bottom of a monitoring well or piezometer

1.4 REFERENCES

SOP No. 002, General Equipment Decontamination

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1.5 REQUIREMENTS AND RESOURCES

The equipment required for measuring static water levels, total well depths, and immiscible layers is as follows:

- Electrical water level indicator
- Interface probe
- PID or FID

2.0 PROCEDURES

This section provides general guidance followed by specific procedures for static water level, total well depth, and immiscible layer measurement.

Techniques for measuring depth to water and depth to the bottom of a monitoring well should be identified in the planning stage of field work. Also at this stage, measuring devices should be chosen, and an individual should be assigned to take and record measurements.

All measurement instruments should be decontaminated before and after use and between measurement locations. Refer to SOP No. 002, General Equipment Decontamination.

Before initiating any measuring activities, the ambient air at a monitoring well head should be monitored for possible emissions of volatile organic compounds. To accomplish this monitoring, a PID or an FID should be used. The health and safety plan for on-site activities should provide action levels and the rationale for selection of either detector.

Appropriate respiratory protection equipment should be worn by the sampling team. Wells should be approached from the upwind side. When opening the monitoring well, the sampling team should systematically survey the inside of the well casing, the area from the casing to the ground, the area from above the well casing to the breathing zone, and the area around the well. Readings for comparison to action levels should be taken not within the well casing but in the breathing zone. If PID or FID readings of volatile organic compounds are above action levels, the sampling team should retreat to a safe area and put on appropriate safety gear. The site-specific health and safety plan should be consulted for action levels.

2.1 STATIC WATER LEVEL MEASUREMENT

The procedure described below should be followed to measure the static water level in a monitoring well or piezometer.

An electric water level indicator is typically used for static water level measurement. The electrical probe of the indicator should be lowered into the monitoring well until the light or sound alarm is activated, indicating that the probe has touched the water surface. The static water level should then be read directly from the indicator to the 0.01-foot fraction. If the monitoring well top is not flush with the ground surface, the distance between the static water level and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth to static water below ground surface. If surveyed elevations are available, they should be used to establish the water level elevation. To ensure measurement accuracy, the probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the values should be averaged. The measurement date and time, individual readings, and the average of the readings should be recorded in a field logbook.

2.2 TOTAL WELL DEPTH MEASUREMENT

The procedure described below should be followed to measure total well depth in a monitoring well or piezometer.

Total well depth measurement can be performed also using an electric water level indicator. The electrical probe of the indicator should be lowered into the monitoring well until resistance is met, indicating that the probe has reached the bottom of the well. The total well depth should then be read directly from the indicator to the 0.01-foot fraction. If the monitoring well top is not flush with the ground surface, the distance between the bottom of the well and the top of the riser pipe should be measured; the height of the riser pipe above ground surface should then be subtracted from the first measurement to determine the depth from ground surface to the bottom of the well. To ensure measurement accuracy, the probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the values should be averaged. The measurement date and time, individual readings, and the average of the readings should be recorded in a field logbook.

2.3 IMMISCIBLE LAYER DETECTION AND MEASUREMENT

The procedure described below should be followed to detect and measure an immiscible layer in a monitoring well.

A light immiscible layer in a monitoring well can be detected by slowly lowering an interface probe to the surface of the water in the well. When the audible alarm sounds, the depth of the probe should be recorded. If the alarm is continuous, a light immiscible layer has been detected. To measure the thickness of this layer, the probe should then be lowered until the alarm changes to an oscillating signal. The oscillating alarm indicates that the probe has reached a water layer. The probe depth at the time the alarm begins oscillating should be recorded as the depth to water. The thickness of the light immiscible layer should then be determined by subtracting the depth at which a continuous alarm occurred from the depth at which the alarm began to oscillate. To ensure measurement accuracy, the interface probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the depths and thicknesses measured should be averaged. The measurement date and time, individual readings for depth and thickness, and average values for depth and thickness should be recorded in a field logbook.

To determine whether a dense immiscible layer is present, the interface probe should be lowered further into the monitoring well. If the alarm changes from an oscillating to a continuous signal, a heavier immiscible layer has been detected, and the probe depth should be recorded at that point. Total well depth obtained in Section 2.2 should be used for calculating the thickness of the dense layer. The dense layer should be calculated by subtracting the depth at which the alarm became continuous from the total well depth. This procedure provides an estimate of the thickness of the dense layer in the monitoring well. To ensure measurement accuracy, the interface probe should be left hanging above the water surface in the monitoring well; a series of three readings should be taken, and the depths and thicknesses measured should be averaged. The measurement date and time, individual readings for depth and thickness, and average values for depth and thickness should be recorded in a field logbook.

STANDARD OPERATING PROCEDURE (SOP) APPROVAL FORM

SULLIVAN INTERNATIONAL GROUP, INC.
ENVIRONMENTAL SOP

ASBESTOS SAMPLING
SOP NO. 014
REVISION NO. 00

Last Reviewed: April 16, 2007

Quality Assurance Approved

April 16, 2007
Date

1.0 BACKGROUND

Asbestos was one of the first hazardous air pollutants regulated under Section 112 of the Clean Air Act (CAA). On March 31, 1971, The U.S. Environmental Protection Agency identified asbestos as a hazardous pollutant, and on April 6, 1973, EPA first promulgated the Asbestos National Emissions Standards for Hazardous Air Pollutants (NESHAP) in 40 Code of Federal Regulation Part 61. This standard operating procedure (SOP) describes the field procedure for taking bulk asbestos samples in order to comply with the asbestos NESHAP regulations.

This SOP is designed to be used as an integral part of a sampling and analysis plan which outlines sampling methods and provides preliminary rationale for sampling locations. Sampling locations may be adjusted in the field based on the screening methods being used and the physical features of the area.

1.1 PURPOSE

Sampling potential asbestos containing material is conducted to determine the presence, type, and amount of asbestos in building materials. Proper bulk-sampling procedures ensure the safety of the individual taking the sample and building occupants.

1.2 SCOPE

This SOP describes procedures for bulk asbestos sampling.

1.3 DEFINITIONS

Asbestos: Any of a member of six fibrous silicate materials that occur naturally in the earth's crust, including Amphiboles and Serpentines (chrysotile, amosite, crocidolite, and fribrous tremolite, anthophylite, actinolite).

Cork Borer: a metal tool use for cutting a hole in potential asbestos-containing materials.

Drop Cloth: A plastic cloth used to protect floors and work surfaces from spills and spatters.

Friable Asbestos Containing Material (ACM): Any material containing more than one percent (1%) asbestos that, when dry, can be crumbled, pulverized or reduced to powder by hand pressure.

High-Efficiency Particulate Air (HEPA) filter cartridges: A cartridge containing a filter manufactured to ensure the highest quality performance and specifications that can filter the finest of dust particles while allowing air to flow thru for breathing.

National Emissions Standards for Hazardous Air Pollutants: Emission standards set by the U.S. Environmental Protection Agency for an air pollutant not covered by National Ambient Air Quality Standards that may cause an increase in fatalities or in serious, irreversible, or incapacitating illness.

1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1985. "Asbestos in Buildings – Simplified Sampling Scheme for Friable Surfacing Materials." EPA 560/5-85-030A.

EPA. 2003. *40 Code of Federal Regulation, Part 763.* July 1.

1.5 REQUIREMENTS AND RESOURCES

The following field equipment is necessary for bulk sampling of potential asbestos containing material (ACM) for determining the presence of asbestos:

- Certified pre-cleaned sample containers with Teflon-lined lids or Ziploc® bags
- Chain of Custody forms
- Cork borer
- Hammer, screwdriver, or pliers
- Ink pens
- Knife with retractable blade
- Powder-free Nitrile gloves
- Respirator with adequate quantities of High-Efficiency Particulate Air (HEPA) filter cartridges
- Sample labels
- Utility knife

2.0 PROCEDURE

To ensure consistency in bulk asbestos sampling, the following procedure will be followed once the sampling locations are identified:

- Don all required personal protective equipment as identified in project health and safety plan, including at a minimum, of a half face respirator.
- Don new and unused disposable (powder-free) Nitrile gloves.
- Place a plastic drop cloth under foot or ladder, when collecting bulk samples from elevated interior locations.

- For samples above the floor level, wet the surface of the material to be sampled by misting water on the sample location, then place sample container or Ziploc® bag at the sample location so any debris will fall into the open collection bag.

- For samples locations at the floor, wet the surface of the material to be sampled by misting water on the sample location.

- Score the material to be sampled with a sharp clean cutting tool (utility knife or cork borer), collecting approximately 1 square inch of material, using a template for accuracy.

- Place the material for sampling into a certified pre-cleaned container with Teflon-lined lid or Ziploc® bag, label for asbestos analysis, and transport as prescribed in SOP 001.

- Encapsulate the sampled surface with appropriate material to prevent release of potentially friable ACM.

- Fold the plastic drop cloth in gently to prevent any potential ACM debris from entering the air and dispose as noted in the work plan.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GENERAL EQUIPMENT DECONTAMINATION

SOP NO. 002

REVISION NO. 2

Last Reviewed: December 1999

Quality Assurance Approved

February 2, 1993

Date

1.0 BACKGROUND

All nondisposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples and to reduce the possibility of cross-contamination.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for decontaminating equipment in the field.

1.2 SCOPE

This SOP applies to decontaminating general nondisposable field equipment. To prevent contamination of samples, all sampling equipment must be thoroughly cleaned prior to each use.

1.3 DEFINITIONS

Alconox: Nonphosphate soap

1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1992. "RCRA Ground-Water Monitoring: Draft Technical Guidance. Office of Solid Waste. Washington, DC. EPA/530-R-93-001. November.

EPA. 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP #2006 (Rev. #0.0, 08/11/94). On-Line Address: http://204.46.140.12/media_resrcs/media_resrcs.asp?Child1=

1.5 REQUIREMENTS AND RESOURCES

The equipment required to conduct decontamination is as follows:

- Scrub brushes
- Large wash tubs or buckets
- Squirt bottles

- Alconox
- Tap water
- Distilled water
- Plastic sheeting
- Aluminum foil
- Methanol or hexane
- Dilute (0.1 N) nitric acid

2.0 PROCEDURE

The procedures below discuss decontamination of personal protective equipment (PPE), drilling and monitoring well installation equipment, borehole soil sampling equipment, water level measurement equipment, and general sampling equipment.

2.1 PERSONAL PROTECTIVE EQUIPMENT DECONTAMINATION

Personnel working in the field are required to follow specific procedures for decontamination prior to leaving the work area so that contamination is not spread off-site or to clean areas. All used disposable protective clothing, such as Tyvek coveralls, gloves, and booties, will be containerized for later disposal. Decontamination water will be containerized in 55-gallon drums.

Personnel decontamination procedures will be as follows:

1. Wash neoprene boots (or neoprene boots with disposable booties) with Liquinox or Alconox solution and rinse with clean water. Remove booties and retain boots for subsequent reuse.

2. Wash outer gloves in Liquinox or Alconox solution and rinse in clean water. Remove outer gloves and place into plastic bag for disposal.

3. Remove Tyvek or coveralls. Containerize Tyvek for disposal and place coveralls in plastic bag for reuse.

4. Remove air purifying respirator (APR), if used, and place the spent filters into a plastic bag for disposal. Filters should be changed daily or sooner depending on use and application. Place respirator into a separate plastic bag after cleaning and disinfecting.

5. Remove disposable gloves and place them in plastic bag for disposal.

6. Thoroughly wash hands and face in clean water and soap.

2.2 DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT DECONTAMINATION

All drilling equipment should be decontaminated at a designated location on-site before drilling operations begin, between borings, and at completion of the project.

Monitoring well casing, screens, and fittings are assumed to be delivered to the site in a clean condition. However, they should be steam cleaned on-site prior to placement downhole. The drilling subcontractor will typically furnish the steam cleaner and water.

After cleaning the drilling equipment, field personnel should place the drilling equipment, well casing and screens, and any other equipment that will go into the hole on clean polyethylene sheeting.

The drilling auger, bits, drill pipe, temporary casing, surface casing, and other equipment should be decontaminated by the drilling subcontractor by hosing down with a steam cleaner until thoroughly clean. Drill bits and tools that still exhibit particles of soil after the first washing should be scrubbed with a wire brush and then rinsed again with a high-pressure steam rinse.

All wastewater from decontamination procedures should be containerized.

2.3 BOREHOLE SOIL SAMPLING EQUIPMENT DECONTAMINATION

The soil sampling equipment should be decontaminated after each sample as follows:

1. Prior to sampling, scrub the split-barrel sampler and sampling tools in a bucket using a stiff, long bristle brush and Liquinox or Alconox solution.
2. Steam clean the sampling equipment over the rinsate tub and allow to air dry.
3. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
4. Containerize all water and rinsate.

5. Decontaminate all pipe placed down the hole as described for drilling equipment.

2.4 WATER LEVEL MEASUREMENT EQUIPMENT DECONTAMINATION

Field personnel should decontaminate the well sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures should be used:

1. Wipe the sounding cable with a disposable soap-impregnated cloth or paper towel.
2. Rinse with deionized organic-free water.

2.5 GENERAL SAMPLING EQUIPMENT DECONTAMINATION

All nondisposable sampling equipment should be decontaminated using the following procedures:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Maintain the same level of protection as was used for sampling.
3. To decontaminate a piece of equipment, use an Alconox wash; a tap water wash; a solvent (methanol or hexane) rinse, if applicable or dilute (0.1 N) nitric acid rinse, if applicable; a distilled water rinse; and air drying. Use a solvent (methanol or hexane) rinse for grossly contaminated equipment (for example, equipment that is not readily cleaned by the Alconox wash). The dilute nitric acid rinse may be used if metals are the analyte of concern.
4. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
5. Containerize all water and rinsate.

REMEDIAL ACTION CONTRACT 2 FOR
REMEDIAL, ENFORCEMENT OVERSIGHT, AND
NON-TIME-CRITICAL REMOVAL ACTIVITIES
IN REGION 5

ATTACHMENT B

PHASE II QUALITY ASSURANCE PROJECT PLAN
MATTHIESSEN AND HEGELER ZINC COMPANY SITE
OPERABLE UNIT 2
LASALLE COUNTY, ILLINOIS

Prepared for
U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604

Date Submitted:	August 21, 2008
EPA Region:	5
Work Assignment No:	032-RICO-B568
Contract No:	EP-S5-06-02
Prepared by:	SulTRAC
Project Manager:	Jennifer Knoepfle
Telephone No:	(312) 443-0550, ext.16
EPA Work Assignment Manager:	Demaree Collier
Telephone No:	(312) 886-0214

CONTENTS

TABLES

FIGURES

ACRONYMS AND ABBREVIATIONS

%D	Percent difference
%R	Percent recovery
µg/L	Microgram per liter
µm	Micrometer
AES	Atomic emission spectroscopy
ASTM	American Society of Testing Materials
bgs	Below ground surface
CA	Corrective action
$CaCO_3$	Calcium carbonate
CADRE	Computer-aided data review
CAS	Chemical Abstract Services
cc	Cubic centimeter
CCV	Continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	Calibration factor
CLP	Contract Laboratory Program
CMS	Carbon molecular sieve
CRL	Central regional laboratory
CRQL	Contract-required quantitation limit
DQI	Data quality indicator
EPA	U.S. Environmental Protection Agency
FID	Flame ionization detector
FIELDS	Field Environmental Decision Support
FS	Feasibility study
FSP	Field sampling plan
GC	Gas chromatography
HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
HCl	Hydrochloric acid
HNO_3	Nitric acid
ICP	Inductively coupled plasma
ID	Identification

ACRONYMS AND ABBREVIATIONS (CONTINUED)

IDPH	Illinois Department of Public Health
IEPA	Illinois Environmental Protection Agency
L/min	Liter per minute
LEGS	Laboratory for Environmental and Geological Studies
LIMS	Laboratory information management system
M&H Site	Matthiessen and Hegeler Zinc Site
MCE	Mixed cellulose ester
MCL	Maximum contaminant level
mg/kg	Milligram per kilogram
mL	Milliliter
mm	Millimeter
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
NaOH	Sodium hydroxide
NC	No criteria
NFG	National Functional Guidelines
NIOSH	National Institute for Occupational Safety and Health
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OU	Operable unit
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PID	Photoionization detector
ppm	Part per million
PQO	Project quality objective
PRG	Preliminary remediation goal
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
QL	Quantitation limit
RAC	Remedial Action Contract
RI	Remedial investigation
RPD	Relative percent difference

ACRONYMS AND ABBREVIATIONS (CONTINUED)

RRF	Relative response factor
RSCC	Regional Sample Control Coordinator
RSD	Relative standard deviation
SAP	Sampling and analysis plan
SOP	Standard operating procedure
SMO	Sample Management Office
SOW	Statement of work
SPLP	Synthetic precipitation leaching procedure
SVOC	Semivolatile organic compound
TAL	Target Analyte List
TBD	To be determined
TCE	Trichloroethene
TCLP	Toxicity characteristic leaching procedure
UFP	Uniform Federal Policy for Implementing Environmental Quality Systems
VOC	Volatile organic compound
WA	Work assignment
WAM	Work assignment manager
XRF	X-ray fluorescence

1.0 INTRODUCTION

SulTRAC has prepared this quality assurance project plan (QAPP) as part of the sampling and analysis plan (SAP) for the Matthiessen and Hegeler Zinc Company (M&H) Site in LaSalle, LaSalle County, Illinois, under the U.S. Environmental Protection Agency (EPA) Response Action Contract (RAC) II for Region 5, Contract No. EP-S5-06-02, Work Assignment (WA) No. 032-RICO-B568. This QAPP is specific to operable unit 2 (OU2) of the M&H Site. The M&H Site is a Superfund Site because of the presence of documented hazardous substances and releases, particularly heavy metals. The SAP consists of the field sampling plan (FSP) (Attachment A) and the QAPP (Attachment B), which are among the site-specific plans to be prepared under the WA in accordance with Task 1 of the EPA statement of work (SOW) (EPA 2008).

This QAPP describes quality assurance (QA) and the quality control (QC) protocols and objectives, methods, and procedures to be performed by SulTRAC during the second field investigation (Phase II) of the remedial investigation/feasibility study (RI/FS) at the OU2 M&H Site. The scope of the QAPP, as outlined in the M&H Site work plan for OU2 (SulTRAC 2008b), has been developed to complete the characterization of contamination begun during the initial field investigation (Phase I) and to delineate the extent of this contamination within the OU2 M&H Site. QAPP scoping information directly related to OU2 was taken from M&H Site fire insurance maps, aerial photographs, architectural drawings, site diagrams, engineering maps, laboratory notebooks, and other assorted documents that describe building construction, operation details, M&H Site processes, and analytical results from the Phase I field investigation conducted from July 16 through November 16, 2007.

This QAPP discusses only Phase II field sampling activities. Section 2.0 of this QAPP discusses the site description and history, and Section 3.0 discusses the QAPP procedures. The QAPP worksheets are presented after Section 3.0. References used to prepare this QAPP are listed after the worksheets, and tables and figures are presented after the list of references.

2.0 SITE DESCRIPTION AND HISTORY

The entire M&H Site, located in LaSalle, LaSalle County, Illinois, occupies about 160 acres inclusive of inactive primary zinc smelting operations and associated abandoned buildings, a rolling mill, and the active Carus Chemical Company and its property (see Figure 1). The M&H Site is bounded by the Little Vermilion River to the north and east and by private residences to the south and west. Tracts of farmland and a limestone quarry are located across the Little Vermilion River north and east of the site, respectively.

The City of LaSalle obtains its drinking water from a cluster of four wells located 0.75 mile south of the M&H Site, and the nearest municipal well also is located about 0.75 mile south of the M&H Site. An abandoned sewer line runs across the property, which serves as a transport mechanism for surface water runoff directly into the Little Vermilion River. A wetland is located approximately 0.5 mile upstream from the M&H Site, and the Illinois River is located approximately 1 mile downstream of the M&H Site. The Lake DePue Fish and Wildlife Area and the Spring Lake Heron Colony are situated about 15 miles downstream of the M&H Site.

2.1 SITE HISTORY

The M&H Site began operations in 1858 when raw materials such as zinc ore and various grades of coal were transported to smelt zinc. A rolling mill was built on site in 1866 to produce zinc sheets. This process included a furnace that used producer gas as fuel, and any sulfur dioxide generated was recovered and converted into sulfuric acid stored in on-site tanks. The M&H Site also had an ammonium sulfate fertilizer plant that operated for a few years during the early 1950s. Coal mining occurred at the M&H Site until 1937, and two mining shafts (one vertical and one horizontal) remain today. Zinc smelting ceased in 1961, and sulfuric acid manufacturing halted in 1968. From 1968 until 1978, when bankruptcy was declared, the facility only performed rolling mill operations. Mr. and Mrs. Fred and Cynthia Carus purchased the 12-acre rolling mill tract in 1980, which became the LaSalle Rolling Mills.

The LaSalle Rolling Mills worked under contract with the United States Mint to generate metal blanks for pennies and operated until 2000, when bankruptcy was declared. In 2003, EPA conducted an emergency removal at the LaSalle Rolling Mills to address cyanide contamination, the old plating line, and various other chemicals and storage tanks that remained after closure of the rolling mill. This removal action is complete. The Carus Chemical Company and Carus Chemical property, which are owned by Mr. Paul Carus, are located south of the rolling mill. The chemical company has been operational since 1915 and mainly produces potassium permanganate.

The M&H Site has been divided into two OUs, OU1 and OU2. As negotiated by a settlement order signed in September 2006, OU1 includes the Carus Chemical Company and property, the Little Vermilion River adjacent to the entire M&H Site, and a large slag and sinter waste pile occupying about 6 acres and 40 to 100 feet deep. OU2 occupies about 140 acres and is identified as the production area of the former zinc smelting and rolling processes and the immediate property surrounding this area. Specifically, OU2 includes the former rolling mill facility, approximately 150 associated former buildings and structures, a shallow slag and sinter pile that heterogeneously covers the former production area of the M&H Site, several abandoned and closed mine shafts, an undeveloped woodland, and surrounding residential areas. Most of the residential area is being investigated by the EPA Field Environmental Decision Support (FIELDS) Team.

2.2 PREVIOUS SITE INVESTIGATIONS

The M&H Site was listed on the National Priorities List (NPL) on September 29, 2003. Two primary sources located on the property were used to score the site for the NPL. The first source is the 6-acre waste pile mostly located on the Carus Chemical Company property of the M&H Site (OU1). This contamination source is addressed under a separate WA (015-RSBD-B568) and will not be further discussed in this QAPP.

The second source is a shallow waste pile composed of sinter and slag heterogeneously deposited throughout the former smelter property included within OU2. The contaminants discovered in the second source appear to be the result of former zinc smelter activities and ancillary operations as described above. Runoff from this shallow sinter and slag pile flows into the Little Vermilion River through natural drainage pathways and manmade conduits. In the central portion of OU2 west of the abandoned railroad are conduits running from an abandoned pump house to the Little Vermilion River as well as drainage that enters an old abandoned and collapsed storm sewer line that runs east-west across the entire width of OU2.

During the November 1991 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) screening site inspection and the December 1993 CERCLA integrated assessment sampling, the Illinois Environmental Protection Agency (IEPA) collected several samples from the two sources. Five samples were collected from the sinter slag cover on OU2. The IEPA also observed a release to surface water during the 1993 screening that was subsequently substantiated through chemical analyses of sediment samples collected from the Little Vermilion River.

The preliminary results of the 2007 Phase I RI show ubiquitous metals contamination across the OU2 M&H Site, namely arsenic, lead, cadmium, copper, mercury, and zinc in soils, debris piles, building materials, surface water, and groundwater. OU2 also contains areas of high polychlorinated biphenyl (PCB) contamination in debris piles and surface and subsurface soils near Building 100, the rolling mill, and the furnaces. Trichloroethene (TCE) contamination is also present in soils and groundwater near the rolling mill at OU2. Polycyclic aromatic hydrocarbons (PAH) were detected ubiquitously at OU2. In addition, asbestos has been detected at concentrations as high as 6.5 percent. Based on these results, OU2 has been fairly well characterized, and the goal of the 2008 Phase II RI is to determine the extent and delineation of contamination at OU2.

Chemicals of interest that have been identified as potentially hazardous to human health and the environment at OU2 are based on the documented investigations discussed above and on information obtained by SulTRAC. These chemicals are listed in the table below.

CHEMICALS OF INTEREST AT OU2

Chemical of Interest	IEPA Assessment Maximum Surface Concentration[1] (mg/kg)	RI/FS Phase I - Maximum Surface Concentration[2,3] (mg/kg)	RI/FS Phase I- Maximum Subsurface Concentration[3,4] (mg/kg)
Cadmium	1,320	7,350	770
Copper	3,650	7,020	2,430
Lead	4,310	51,900	62,600
Zinc	71,200	408,000	158,000
Arsenic	36	812	528
Mercury	Unknown	154	143
PCBs	Unknown	150	18
Asbestos	Unknown	6.5%	No detections at depth
TCE	Unknown	0.01	120

Notes:

IEPA Illinois Environmental Protection Agency
mg/kg Milligram per kilogram
OU Operable Unit
PCB Polychlorinated biphenyl
RI/FS Remedial investigation/Feasibility study
TCE Trichloroethene

1 Results are based on five samples collected in 1993 (IEPA 1994).
2 Results are based on 250 samples collected in July and August 2007 (SulTRAC 2008b).
3 Surface sample depths are 0 to 2 feet below ground surface.
4 Subsurface depths range from 8 to 12 feet below ground surface.

3.0 QUALITY ASSURANCE PROJECT PLAN PROCEDURES

This QAPP presents procedures that will be used to ensure the quality of data generated for the OU2 M&H Site. The QAPP provides a framework for how environmental data will be collected to achieve specific project objectives and describes procedures that will be implemented to obtain data of known and adequate quality. This QAPP was prepared in accordance with the EPA's "Uniform Federal Policy for Implementing Environmental Quality Systems" (UFP) (EPA 2005a).

During Phase I, SulTRAC conducted soil, solids (building materials, debris piles, and waste materials), surface water, and groundwater sampling activities. The results of the sampling activities were evaluated to determine the nature of contamination and to identify the contamination sources at the OU2 M&H Site. SulTRAC collected samples from 196 borings, 10 former and/or existing building structures, 55 debris/waste piles, 19 previously installed monitoring wells, and seven surface water locations. Samples were analyzed for volatile organic compounds (VOC), semivolatile organic compounds (SVOC), PCBs, pesticides, target analyte list (TAL) metals (including mercury) and cyanide, and asbestos. During Phase I, SulTRAC also conducted ecological investigations, including a wetland and habitat delineation/function and value assessment, wildlife observations, and identification of endangered species and other species of special concern.

Prior to Phase II field sampling activities, SulTRAC will conduct asbestos air sampling inside the rolling mill. This air monitoring will be conducted at two locations during an active day of Fred Carus's warehousing business, which operates out of this facility. Inside the rolling mill, backer-board is warehoused and distributed with daily pickups of the product by tractor trailer vehicles. Two additional samples will be collected from two downwind locations in the former main industrial plant area where analytical results indicated the highest detections of asbestos in soils during Phase I. Air samples will be analyzed for asbestos by a subcontracted laboratory as identified in Worksheets #3 and #7.

During the Phase II field investigation at OU2, which is anticipated to start in July 2008, SulTRAC will collect samples from approximately 60 soil boring locations, 50 existing and former building structures, 8 surface water locations, and 19 Phase I and 17 Phase II (newly installed) monitoring wells to supplement field work sampling conducted during Phase I. Additionally, the EPA FIELDS team and SulTRAC will conduct x-ray fluorescence (XRF) screening and analytical sampling of approximately 50 surface soil samples at the OU2 M&H Site. Soil samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, TAL metals (including mercury) and cyanide, toxicity characteristic leaching procedure (TCLP) metals, and asbestos. Specialty analyses to examine geochemistry will include metals synthetic precipitation leaching

procedure (SPLP) analysis. Surface water and groundwater samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, and TAL metals (including mercury) and cyanide.

Also during the Phase II investigation, SulTRAC will conduct ecological (bioavailability sampling) and biological (bioassessibility sampling) investigations. Bioavailability sampling will include aboveground and belowground paired vegetation samples, soil invertebrate sampling (worms), and surface soil and soil/sinter/slag samples collected from various areas of ecological interest at OU2. SulTRAC will attempt to collect *in situ* samples, but if enough biomass cannot be collected, the corresponding soil/sinter/slag materials of interest will be collected and sent to a laboratory for *in vivo* growth and analysis. This QAPP discusses this contingency sampling (*in vivo* growth and analysis), along with laboratory procedures and subcontracting information. Bioassessibility sampling will include soil, sinter, and slag sample collection throughout OU2. The results of the sampling activities will be evaluated to further characterize contamination sources and to delineate the extent of contamination at the OU2 M&H Site.

QAPP WORKSHEET #1
TITLE AND APPROVAL PAGE

Quality Assurance Project Plan for Remedial Investigation/Feasibility Study, Matthiessen and Hegeler Zinc Company Site, Operable Unit 2, LaSalle County, Illinois
Document Title

SulTRAC
Lead Organization

Lea Cole and Jennifer Knoepfle SulTRAC
Preparer's Name and Organizational Affiliation

125 South Wacker Drive, Suite 1180, Chicago IL 60640; (312) 443-0550; lcole@onesullivan.com and jknoepfle@onesullivan.com
Preparer's Address, Telephone Number, and E-mail Address

May 30, 2008 (original), August 21, 2008 (revision 1)
Preparation Date (Day/Month/Year)

Jennifer Knoepfle — 8/21/08
SulTRAC Project Manager — Signature/Date

John Dirgo — 8/21/08
SulTRAC QA Officer — Signature/Date

Approval Signatures:

Signature/Date

Demaree Collier, Work Assignment Manager
Printed Name/Title

Approval Authority
Other Approval Signatures:

Signature/Date

Warren Layne, QAPP Reviewer
Printed Name/Title

QAPP WORKSHEET #2
QAPP IDENTIFYING INFORMATION

1. Identify guidance used to prepare QAPP:
"Uniform Federal Policy for Implementing Environmental Quality Systems" (UFP) (EPA 2005a) and "EPA Guidance for Quality Assurance Project Plans" (EPA 2002)

2. Identify regulatory program:
CERCLA

3. Identify approval entity: EPA Region 5

4. Indicate whether the QAPP is a generic or project-specific QAPP: Project-specific

5. List dates of scoping sessions that were held: December 28, 2006; March 10, 2008

6. List dates and titles of QAPP documents written for previous work site, if applicable:

Title	Approval Date
"Phase I Quality Assurance Project Plan, Matthiessen and Hegeler Zinc Company Site, Operable Unit 2, LaSalle County, Illinois"	May 31, 2007

7. List organizational partners (stakeholders) and connection with lead organization:
EPA Region 5, SulTRAC, Illinois Environmental Protection Agency (IEPA)

8. List data users: EPA Region 5, SulTRAC, IEPA

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below: Not applicable

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

QAPP WORKSHEET #2 (CONTINUED)
QAPP IDENTIFYING INFORMATION

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2.2.1 Document Control Format	QAPP Identifying Information	2
2.2.2 Document Control Numbering System		
2.2.3 Table of Contents		
2.2.4 QAPP Identifying Information		
2.3 - Distribution List and Project Personnel Sign-Off Sheet		
2.3.1 Distribution List	Distribution List	3
2.3.2 Project Personnel Sign-Off Sheet	Project Personnel Sign-Off Sheet	4
2.4 - Project Organization		
2.4.1 Project Organization Chart	Project Organization Chart	5
2.4.2 Communication Pathways	Communication Pathways	6
2.4.3 Personnel Responsibilities and Qualifications	Personnel Responsibilities and Qualifications	7
2.4.4 Special Training Requirements and Certification	Special Training Requirements and Certification	8
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	Project Scoping Session Participants Sheet	
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QAPP WORKSHEET #2 (CONTINUED)
QAPP IDENTIFYING INFORMATION

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QAPP WORKSHEET #2 (CONTINUED)
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QAPP WORKSHEET #2 (CONTINUED)
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QAPP WORKSHEET #3
DISTRIBUTION LIST

(UFP QAPP Section 2.3.1)
List individuals who received copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments.

QAPP Recipient	Title	Organization	Telephone Number	E-mail Address
Demaree Collier	Work Assignment Manager (WAM)	EPA Region 5	(312) 886-0214	collier.demaree@epa.gov
Warren Layne	QAPP Reviewer	EPA Region 5	(312) 886-7336	layne.warren@epa.gov
Thomas Williams	Project Manager	IEPA	(312) 886-0814	william.thomas@illinois.gov
Jennifer Knoepfle	Project Manager	SulTRAC	(312) 443-0550, ext. 16	jknoepfle@onesullivan.com
Tracey Koach	Co-Field Team Leader	SulTRAC	(312) 443-0550, ext. 11	tkoach@onesullivan.com
Cheryl Gorman	Co-Field Team Leader and Sample Custodian	SulTRAC	(312) 443-0550, ext. 17	cgorman@onesullivan.com
Lea Cole	Project Scientist and Sample Custodian	SulTRAC	(312) 443-0550, ext. 15	lcole@onesullivan.com
Robert Kondreck	Project Scientist	SulTRAC	(312) 201-7479	robert.kondreck@ttemi.com
Richard Baldino	Project QA Manager	SulTRAC	(847) 494-2685	rbaldino@onesullivan.com
John Dirgo	QA/QC Officer	SulTRAC	(312) 201-7765	john.dirgo@ttemi.com
William Earle	Analytical Coordinator	SulTRAC	(312) 443-0550, ext. 12	wearle@onesullivan.com
David Homer	Ecological Risk Assessor	SulTRAC	(816) 412-1762	david.homer@ttemi.com
Eric Morton	Human Health Risk Assessor	SulTRAC	(312) 201-7797	eric.morton@ttemi.com
Larry Horn	Business Development Manager	STAT Analysis Corporation (for asbestos analyses)	(312) 733-0551	LHorn@STATAnalysis.com
Subcontractors	Drillers/Geoprobers/ Surveyors	TBD	TBD	TBD

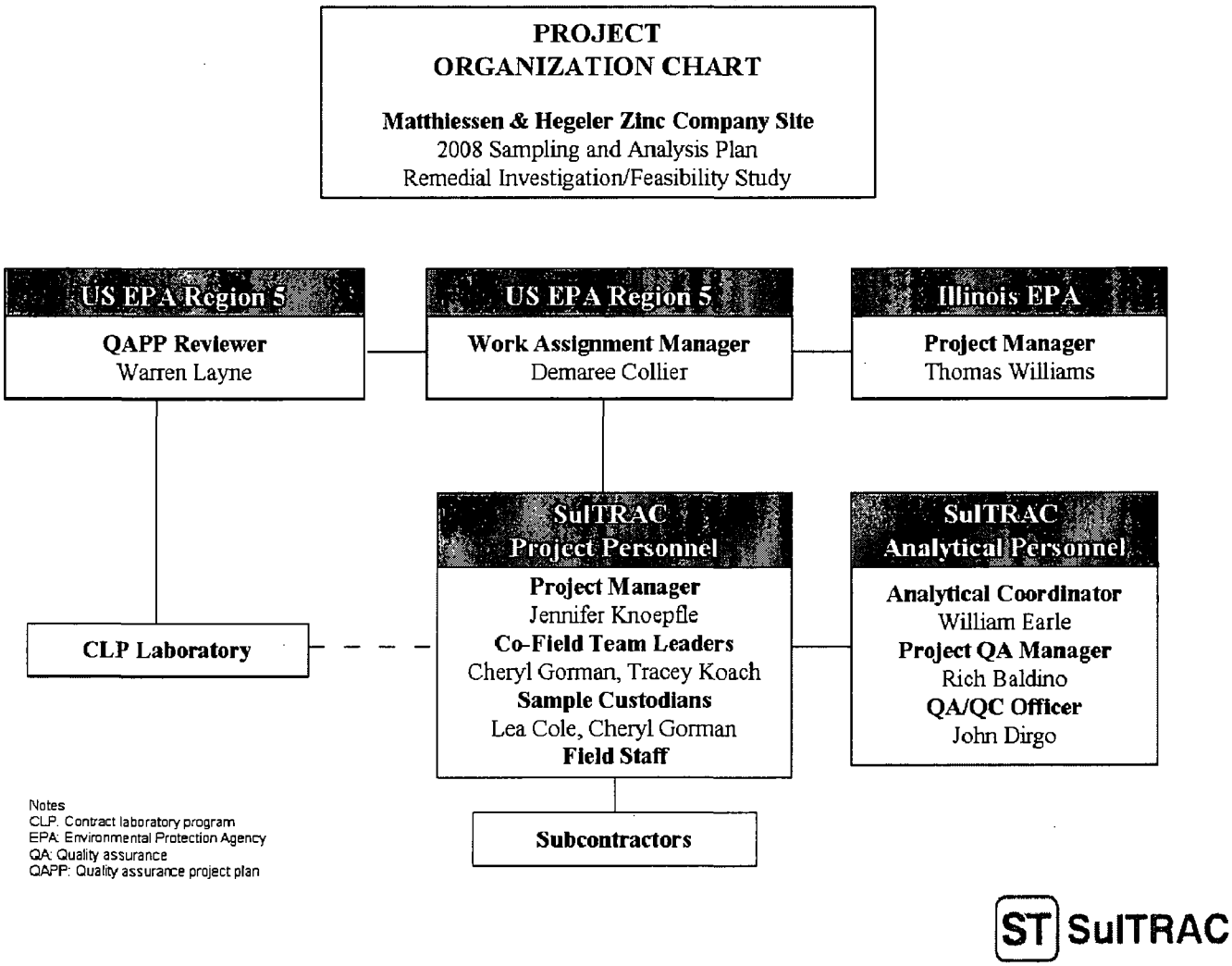
QAPP WORKSHEET #4
PROJECT PERSONNEL SIGN-OFF SHEET

(UFP QAPP Section 2.3.2)
Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to central project file.

Project Personnel	Organization	Title	Telephone No.	Signature	Date QAPP Read
Jennifer Knoepfle	SulTRAC	Project Manager	(312) 443-0550, ext. 16		8/21/08
William Earle	SulTRAC	Analytical Coordinator	(312) 443-0550, ext. 11		8/21/08
Richard Baldino	SulTRAC	Project QA Manager	(847) 494-2685		5/15/08
John Dirgo	SulTRAC	QA/QC Officer	(312) 201-7765		5/30/08
Lea Cole	SulTRAC	Project Scientist and Sample Custodian	(312) 443-0550, ext 15		
Cheryl Gorman	SulTRAC	Co-Field Team Leader and Sample Custodian	(312) 443-0550, ext 17		
Tracey Koach	SulTRAC	Co-Field Team Leader	(312)443-0550, ext 11		
Larry Horn	STAT Analysis Corporation (for asbestos analyses)	Business Development Manager	(312) 733-0551		
Drilling Subcontractor	TBD	TBD	TBD		
Geoprobe Subcontractor	TBD	TBD	TBD		
Surveyor Subcontractor	TBD	TBD	TBD		

QAPP WORKSHEET #5
PROJECT ORGANIZATION CHART

(UFP QAPP Section 2.4.1)
Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names of project managers for each organization.



ST SulTRAC

QAPP WORKSHEET #6
COMMUNICATION PATHWAYS

(UFP QAPP Section 2.4.2)
Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modification to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

Communication Drivers	Responsible Entity	Name	Telephone No.	Procedure (Timing, Pathways, etc.)
Point of contact with EPA WAM	Project Manager	Jennifer Knoepfle	(312) 443-0550, ext. 16	Jennifer Knoepfle will forward all materials and information about the project to Demaree Collier.
Manage all project phases	Project Manager	Jennifer Knoepfle	(312) 443-0550, ext. 16	Communicate information to project team (including subcontractors) on a timely basis. Notify EPA WAM by telephone or e-mail of any significant issues. Direct field team and facilitate communication with analytical coordinator. Delivery of all CLP data packages to project QA manager for final review of validation.
Daily field progress report	Field Team Leaders	Tracey Koach Cheryl Gorman	313-910-2589 312-350-0865	Conduct specific field investigation tasks, direct field activities of subcontractors, and provide daily communication with project manager and sample custodian.
Manage field sample organization and delivery to Contract Laboratory Program (CLP)	Sample Custodian	Lea Cole Cheryl Gorman	312-363-7963 312-350-0865	Ensure field staff is collecting samples in proper containers, observing holding times, and properly packaging and preparing samples for shipment. Coordinate daily with analytical coordinator concerning sample quantities and delivery locations and dates. Communicate daily with field staff and project manager regarding any issues and developments.
Point of contact with EPA Region 5 Regional Sample Control Coordinator (RSCC)	Analytical Coordinator	William Earle	(312) 443-0550, ext. 12	Contact the RSCC before each sampling event to schedule CLP laboratory services. Notify sample custodian and project manager of any CLP issues or developments. Track all CLP data deliveries. Notify project manager and forward data to her.

QAPP WORKSHEET #6 (CONTINUED)
COMMUNICATION PATHWAYS

Communication Drivers	Responsible Entity	Name	Telephone No.	Procedure (Timing, Pathways, etc.)
Release of Analytical Data	SulTRAC Project QA Manager	Richard Baldino	(847) 494-2685	No analytical data can be released until validation is completed and Richard Baldino has reviewed and approved the release.
Report of laboratory data quality issues	Laboratory QA Officer	TBD	TBD	All QA/QC issues with project field samples will be reported by the laboratory QA officer to the RSCC.

QAPP WORKSHEET #7
PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

(UFP QAPP Section 2.4.3)
Identify project personnel associated with each organization, contractor, and subcontractor participating in responsible roles. Include data users, decision-makers, project managers, QA officers, project contacts for organizations involved in the project, project health and safety officers, geotechnical engineers and hydrogeologists, field operation personnel, analytical services, and data reviewers. Identify project team members with an asterisk (*).

Name	Title	Organization/ Affiliation	Responsibilities	Education and Experience Qualifications
Jennifer Knoepfle*	Project Manager	SulTRAC	Manages project; coordinates between lead agency and subcontractor; coordinates CLP data deliverables from analytical coordinator to project QA manager; manages field staff	Ph.D. Earth and Environmental Sciences, Hydrogeologist, 6 years of experience
Tracey Koach*	Field Team Leader	SulTRAC	Supervises field sampling and coordinates all field activities; daily reporting to project manager while conducting field activities	B.A. Environmental Studies, 16 years of experience
Cheryl Gorman*	Field Team Leader and Sample Custodian	SulTRAC	Supervises field sampling and coordinates all field activities; daily reporting to project manager while conducting field activities; implements field plan; verifies sample processing, packaging, and shipping	B.S. Earth and Environmental Science, 4 years of experience
Lea Cole*	Project Scientist Sample Custodian	SulTRAC	Prepares QAPP; implements field plan; verifies sample processing, packaging, and shipping	M.S. Biology and Geology, 3 years of experience
Richard Baldino*	Project QA Manager	SulTRAC	QA/QC oversight	M.S. Water Chemistry, Senior Chemist, 15 years of experience
John Dirgo	QA/QC Officer	SulTRAC	QA/QC oversight	Sc.D. Environmental Science and Physiology, 28 years of experience

QAPP WORKSHEET #7 (CONTINUED)
PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

Name	Title	Organization/ Affiliation	Responsibilities	Education and Experience Qualifications
William Earle*	Analytical Coordinator	SulTRAC	Coordinates sample scheduling; verifies sample chain of custody; reviews computer-aided data review (CADRE) results and data from subcontracted laboratories; notifies sample custodian and project manager of any issues or developments	B.S. Civil Engineering, Professional Engineer, 17 years of experience
Robert Kondrek*	Technical Staff	SulTRAC	Implements field plan	B.S. Geology, 4 years of experience
TBD	Drillers/Geoprobers	TBD Subcontractor	Provides subsurface drilling and sampling	TBD
TBD	Surveyors	TBD Subcontractor	Provides outlined surveys of entire OU2 site	TBD
Larry Horn	Business Development Manager	STAT Analysis Corporation	Performs asbestos analysis of air, soil, and solid samples	Chemists, TBD
TBD	Subcontracted Laboratory	TBD Subcontractor	Performs bioavailability sample tests	TBD
John Drexler	Subcontracted Laboratory	Laboratory for Environmental and Geological Studies (LEGS) – University of Colorado	Performs relative bioassessibility leaching procedure	TBD
John Crivellone	Security	Illinois Security Services	Provides security after hours during intrusive field activities	Licensed company, 20 years experience.

QAPP WORKSHEET #8
SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

(UFP QAPP Section 2.4.4)
Provide the following information for those projects requiring personnel with specialized training. Attach training records and/or certificates to the QAPP or note their location.

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates[1]
Field Staff	40-hour or 8-hour refresher - OSHA HAZWOPER training	Various	Various	SulTRAC	SulTRAC	Corporate human resources office
Field Staff	XRF training	EPA	TBD	SulTRAC	SulTRAC	Chicago Office
Drillers/ Geoprobers	40-hour OSHA HAZWOPER training	TBD	TBD	Drillers/Geoprobers	TBD	As noted in subcontract agreement – corporate human resources office

Notes:

HAZWOPER Hazardous Waste Operations and Emergency Response Standard
OSHA Occupational Safety and Health Administration

QAPP WORKSHEET #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

(UFP QAPP Section 2.5.1)
Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project.

Project Name	RI/FS for M&H Site, OU2	Site Name	M&H Site
Projected Date(s) of Sampling	July 2008 through December 2009	Site Location	La Salle County, Illinois
Project Manager	Jennifer Knoepfle		
Date of Session	March 10, 2008		
Scoping Session Purpose:	Define scope of project		

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Demaree Collier	WAM	EPA Region 5	(312) 886-4071	Collier.Demaree@epa.gov	WAM
Jennifer Knoepfle	Project Manager	SulTRAC	(312) 443-0550 ext 16	jknoepfle@onesullivan.com	Project Manager
Ron Riesing	Program Manager	SulTRAC	(312) 201-7722	Ronald.Riesing@ttemi.com	Program Manager

Comments/Decisions: During this meeting, it was decided that WA No. 032-RICO-B568 will cover the Phase II field investigation of the RI/FS at the OU2 M&H Site. Based on the WA duration, the Phase II field investigation will include a total of seven quarters of groundwater sampling, of which six of the seven events will include wells and piezometers installed during both Phase I and Phase II. Specifically, Phase II includes Tasks 1 through 7, Tasks 9 though 13, and Task 15 of the SOW for the WA.

QAPP WORKSHEET #10
PROBLEM DEFINITION

(UFP QAPP Section 2.5.2)
Clearly define the problem and the environmental questions that should be answered for the current investigation and develop the project decision "If..., then..." statements in the QAPP, linking data results with possible actions. The prompts below are meant to help the project team define the problem. They are not comprehensive.

The problem to be addressed by the project: The preliminary results of the 2007 initial field investigation (Phase I) RI show ubiquitous metals contamination across the entire OU2 M&H Site, namely arsenic, lead, cadmium, copper, mercury, and zinc in soils, debris piles, building materials, surface water, and groundwater. OU2 also contains areas of high PCB contamination in debris piles and surface and subsurface soils near Building 100, the rolling mill, and the furnaces. TCE contamination is present in soils and groundwater near the rolling mill at OU2. PAHs were detected ubiquitously at OU2. In addition, asbestos has been detected at concentrations as high as 6.5 percent. This project intends to complete the characterization of contamination begun during Phase I and to delineate the extent of this contamination within the OU2 M&H Site.
The environmental questions being asked: What is the extent of contamination at the OU2 M&H Site?
Observations from any site reconnaissance reports: During the November 1991 CERCLA screening site inspection and the December 1993 CERCLA integrated assessment sampling, IEPA collected several samples from OU1 and OU2. The IEPA also observed a release to surface water during the 1993 screening that was subsequently substantiated through chemical analyses of sediment samples collected from the Little Vermilion River. The chemicals of concern have been identified as potentially hazardous to human health and safety at the M&H Site within OU2.
A synopsis of secondary data or information from site reports: See Worksheet #13
The possible classes of contaminants and the affected matrices: Asbestos air monitoring will be conducted inside the rolling mill and at two additional downwind locations in the former main industrial plant area where analytical results indicated the highest detections of asbestos in soils during Phase I. Soil and building material samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, TAL metals (including mercury) and cyanide, TCLP metals, SPLP metals, and asbestos. Surface water and groundwater samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, TAL metals (including mercury) and cyanide. Additionally, surface water will be sampled and analyzed for total hardness and dissolved (filtered) metals.
Project decision conditions ("If..., then..." statements): If the RI/FS results reveal that contamination at the OU2 M&H Site poses an unacceptable risk to human health and/or the environment, then a remedial action will be implemented. If contamination characterization is localized on the OU2 property based on the biased approach, then Phase II activities will consist of additional sampling involving nonbiased (gridded XRF screening) and data gap sampling.

QAPP WORKSHEET #11
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

(UFP QAPP Section 2.6.1)
Use this worksheet to develop PQOs in terms of type, quantity, and quality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List the PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only; however, they are neither inclusive nor appropriate for all projects.

Who will use the data: EPA Region 5 and SulTRAC will use the data.
What will the data be used for? During the Phase II field investigation, the data will be used to further characterize contamination sources as well as delineate the extent of contamination at the OU2 M&H Site. Data from both the Phase I and Phase II field investigations will be used to conduct a risk assessment for the entire M&H Site and to evaluate remedial alternatives as part of the FS.
What type of data are needed (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)? Air, soil (surface and subsurface), building material, surface water, and groundwater samples will be collected from the OU2 M&H Site. Air samples will be collected during an 8-hour sampling period prior to Phase II field sampling. Soil samples will be collected from borings and as surface grab samples. Building materials will be collected as grab samples from existing building structures and residual material piles from collapsed and demolished buildings. Surface water samples will be collected from eight locations of known areas of surface water documented on site. Groundwater samples will be collected from groundwater monitoring wells. Field screening instruments will include (1) an Innov-X XRF analyzer to detect metals in soils, (2) a PCB/chloride analyzer to confirm PCBs in soils near Building 100, (3) a photoionization detector (PID) to screen all groundwater and soil boring samples, and (4) a water quality meter to monitor all groundwater parameters during sampling. Additionally, soil, sinter, and slag samples will be collected from throughout OU2 for bioassessibility testing. Objectives of the baseline ecological risk assessment are to gain an understanding of the potential uptake of site-specific contaminants by native plants and to provide site-specific information on the potential movement of contaminants within the food chain. Therefore, SulTRAC will attempt to collect six vegetation sample pairs (12 samples) consisting of an aboveground sample and an underground sample. SulTRAC will also attempt to collect approximately 75 earthworms at five locations within each of the four defined on-site habitat areas. As a contingency, 10 soil samples will be collected from each of the four identified habitats at the M&H Site. These soil samples will be used in 28-day bioavailability testing using earthworms and lettuce seedlings at a subcontracted laboratory that specializes in these types of bioavailability tests and tissue analyses.
How "good" do the data need to be in order to support the environmental decision? Ultimately, the data need to allow full assessment of the nature and extent of contamination in the soil/solid, water, and biota samples collected by SulTRAC. The data also need to be validated and used to support risk assessment and the evaluation of remedial alternatives.

QAPP WORKSHEET #11 (CONTINUED)
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

How much data are needed (number of samples for each analytical group, matrix, and concentration)? SulTRAC will collect 4 air samples (two inside the rolling mill and two in the main plant area); 120 samples from 60 soil borings; 50 building material samples; 8 surface water samples; and groundwater samples from 19 Phase I monitoring wells for the June 2008 groundwater event and from 36 monitoring wells (including 17 newly installed Phase II monitoring wells) thereafter for the seven quarters of groundwater sampling events scheduled during the Phase II field investigation. In addition, QC samples will be collected and analyzed, including duplicates, matrix spikes (MS), matrix spike duplicates (MSD), and trip blanks.
Where, when, and how should the data be collected/generated? Phase II sampling activities will take place during Summer and Fall 2008 at the OU2 M&H Site. Prior to any intrusive field sampling, four locations will be sampled for asbestos, two areas inside the rolling mill and two areas in the main plant. Samples will be collected from 60 soil boring locations (two sampled depth intervals each), 50 building structures, 8 surface water locations, and 19 Phase I monitoring wells and 17 newly installed Phase II monitoring wells. Soil borings at 10 of the 60 soil sampling locations will be advanced to a depth greater than 12 feet below ground surface (bgs) depending on PCB concentrations in the area of known PCB contamination at 12 feet bgs. All soil borings will be advanced by the Geoprobe®. All monitoring wells will be installed using rotosonic or hollow-stem auger drilling techniques. All intrusive work will be performed by subcontractors with a SulTRAC geologist. SulTRAC anticipates hiring subcontractors to perform monitoring well and piezometer installation, direct-push technology (Geoprobe®) soil sampling, site surveying, site security, and site trailer mobilization.
Who will collect and generate the data? SulTRAC will collect the samples discussed herein. A subcontracted laboratory will analyze air, soil, and solid samples for asbestos. A laboratory from the EPA CLP will analyze soil, building material, groundwater, and surface water samples for VOCs, SVOCs, PCBs, pesticides, and TAL metals (including mercury) and cyanide. Additionally, a laboratory from the EPA CLP will analyze soil samples (from the main plant area) for TCLP metals and SPLP metals, and building materials samples for TCLP metals. A modified analysis will need to be ordered for building materials because of their overall size -- it will be difficult and near impossible to grind and homogenize stone, brick, and wood samples in the field, and the CLP laboratories will need to perform this process. Another modified analysis will need to be ordered to include hardness analysis for surface water samples by the CLP laboratories. All modified analyses requests will be submitted 3 weeks in advance to the EPA Sample Management Office (SMO). SulTRAC also anticipates submitting vegetation and soil invertebrate and/or soil/sinter/slag samples to a subcontracted laboratory for bioavailability and bioassessibility testing.
How will the data be reported? Data will be reported by the CLP laboratory using standard CLP data reporting techniques. Data will be reported in electronic and hard-copy form. Subcontracted laboratory data will be reported by the subcontracted laboratory using standard data reporting techniques. SulTRAC is responsible for conducting asbestos, bioassessibility, and bioavailability validation of the analytical data generated by the subcontracted laboratory.

QAPP WORKSHEET #11 (CONTINUED)
PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

How will the data be archived? Electronic and hard copies of CLP analytical data will be archived by the CLP laboratory. Electronic and hard copies of subcontracted laboratory data will be archived by the SulTRAC analytical coordinator. Field data (notebooks, sampling sheets, etc.) will be maintained at SulTRAC's Chicago office. SulTRAC will also provide 10-year data storage.

QAPP WORKSHEET #12
MEASUREMENT PERFORMANCE CRITERIA TABLE

(UFP QAPP Section 2.6.2)
Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQIs), measurement performance criteria (MPC) (percent recovery (% R), and relative percent difference (% RPD), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for a specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

Matrix	Soil/Solids[1]
Analytical Group[2]	Volatile Organic Analysis (VOA)/CLP
Concentration Level	Low concentration

Sampling Procedure[3]	**Analytical Method SOP[4]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-3, S-4, S-5, S-6	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias-Contamination	VOC < QL	Trip blank	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias-Contamination	VOC < QL	Rinsate blank	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/Bias	1,1-Dichloroethene: 59-172 %R TCE: 62-137 %R Benzene: 66-142 %R Toluene: 59-139 %R Chlorobenzene: 60-133 %R	MS/MSD	S & A
S-3, S-4, S-5, S-6	A-1	Precision	1,1-Dichloroethene: 22% RPD TCE: 24% RPD Benzene: 21% RPD Toluene: 21% RPD Chlorobenzene: 21% RPD	MS/MSD	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	VOA/CLP
Concentration Level	Low concentration

Sampling Procedure[3]	Analytical Method SOP[4]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-3, S-4, S-5, S-6	A-1	Accuracy	Vinyl chloride-d_3: 68-122 %R Chloroethane-d_5: 61-130 %R 1,1-Dichloroethene-d_2: 45-132 %R 2-Butanone-d_5: 20-182 %R Chloroform-d: 72-123 %R 1,2-Dichloroethane-d_4: 79-122 %R Benzene-d_6: 80-121 %R 1,2-Dichloropropane-d_6: 74-124 %R Toluene-d_8: 78-121 %R 1,1,2,2-Tetrachloroethane-d_2: 56-161 %R Trans-1,3-Dichloropropene-d_4: 72-130 %R 2-Hexanone-d_5: 17-184 %R 1,4-Dioxane-d_8: 50-150 %R 1,2-Dichlorobnzene-d_4: 70-131 %R	Deuterated monitoring compounds	A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias-Contamination	VOC < QL	Method blank	A
S-3, S-4, S-5, S-6	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	Semivolatile Organic Analysis (SVOA)/CLP
Concentration Level	Medium concentration

Sampling Procedure[3]	**Analytical Method SOP[4]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-3, S-4, S-5, S-6	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias-Contamination	SVOC < QL	Rinsate blank	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/Bias	Phenol-d_5: 26-90 %R 2-Chlorophenol: 25-102 %R N-Nitroso-di-n-propylamine: 41-126 %R 4-Chloro-3-methylphenol: 26-103 %R Acenaphthene: 31-137 %R 4-Nitrophenol: 11-114 %R 2,4-Dinitrotoluene: 28-89 %R Pentachlorophenol: 17-109 %R Pyrene: 35-142 %R	MS/MSD	S & A
S-3, S-4, S-5, S-6	A-1	Precision	Phenol: 35% RPD 2-Chlorophenol: 50% RPD N-Nitroso-di-n-propylamine: 38% RPD 4-Chloro-3-methylphenol: 33% RPD Acenaphthene: 19% RPD 4-Nitrophenol: 50% RPD 2,4-Dinitrotoluene: 47% RPD Pentachlorophenol: 47% RPD Pyrene: 36% RPD	MS/MSD	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	Semivolatile Organic Analysis (SVOA)/CLP
Concentration Level	Medium concentration

Sampling Procedure[3]	**Analytical Method SOP[4]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-3, S-4, S-5, S-6	A-1	Accuracy	Phenol-d_5: 17-103 %R Bis(2-Chloroethyl)ether-d_8: 12-98 %R 2-Chlorophenol-d_4: 13-101 %R 4-Methylphenol-d_8: 8-100 %R Nitrobenzene-d_5: 16-103 %R 2-Nitrophenol-d_4: 16-104 %R 2,4-Dichlorophenol-d_3: 23-104 %R 4-Chloroaniline-d_4: 1-145 %R Dimethylphthalate-d_6: 43-111 %R Acenaphthylene-d_8: 20-97 %R 4-Nitrophenol-d_4: 16-166 %R Fluorene-d_{10}: 40-108 %R 4,6-Dinitro-2-methylphenol-d_2: 1-121 %R Anthracene-d_{10}: 22-98 %R Pyrene-d_{10}: 51-120 %R Benzo(a)pyrene-d_{12}: 43-111 %R	Deuterated monitoring compounds	A
S-3, S-4, S-5, S-6	A-1	Accuracy/Bias-Contamination	SVOC < QL	Method blank	A
S-3, S-4, S-5, S-6	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S&A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	PCB/CLP
Concentration Level	Low concentration

Sampling Procedure[3]	Analytical Method SOP[4]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-3, S-4, S-5, S-6, S-18	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-3, S-4, S-5, S-6, S-18	A-1	Accuracy/ Bias-Contamination	PCB < QL	Rinsate blank	S & A
S-3, S-4, S-5, S-6, S-18	A-1	Accuracy/Bias	Aroclor-1016: 29-135 %R Aroclor-1260: 29-135 %R	MS/MSD	S & A
S-3, S-4, S-5, S-6, S-18	A-1	Precision	Aroclor-1016: 15% RPD Aroclor-1260: 20% RPD	MS/MSD	S & A
S-3, S-4, S-5, S-6, S-18	A-1	Accuracy	Decachlorobiphenyl: 30-150 %R	Surrogate spike	A
S-3, S-4, S-5, S-6, S-18	A-1	Accuracy/ Bias-Contamination	PCB < QL	Method blank	A
S-3, S-4, S-5, S-6, S-18	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	Pesticide/CLP
Concentration Level	Low concentration

Sampling Procedure[3]	Analytical Method SOP[4]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-3, S-4, S-5, S-6	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias- Contamination	Pesticides < QL	Rinsate blank	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy/Bias	Gamma-BHC: 46-127 %R Heptachlor: 35-130 %R Aldrin: 34-132 %R Dieldrin : 31-134 %R Endrin : 42-139 %R 4,4'-DDT : 23-134 %R	MS/MSD	S & A
S-3, S-4, S-5, S-6	A-1	Precision	Gamma-BHC: 50% RPD Heptachlor: 31% RPD Aldrin: 43% RPD Dieldrin: 38% RPD Endrin: 45% RPD 4,4'-DDT: 50% RPD	MS/MSD	S & A
S-3, S-4, S-5, S-6	A-1	Accuracy	Tetrachloro-m-xylene: 30-150 %R	Surrogate spike	A
S-3, S-4, S-5, S-6	A-1	Accuracy/ Bias- Contamination	Pesticide < QL	Method blank	A
S-3, S-4, S-5, S-6	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	TAL Metals, Mercury, Cyanide/CLP
Concentration Level	Multi-concentration

Sampling Procedure[3]	**Analytical Method SOP[4]**	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-3, S-4, S-5, S-6, S-7, S-16, S-20	A-2, A-3, A-4, A-5,	Precision	RPD ≤ 70%	Field duplicate	S & A
S-3, S-4, S-5, S-6, S-7, S-16, S-20	A-2, A-3, A-4, A-5,	Accuracy/ Bias-Contamination	Metal < QL	Rinsate blank	S & A
S-3, S-4, S-5, S-6, S-7, S-16, S-20	A-2, A-3, A-4, A-5	Accuracy/Bias	All metals: 75-125 %R	MS	A
S-3, S-4, S-5, S-6, S-7, S-16, S-20	A-2, A-3, A-4, A-5,	Precision	All metals: < 20% RPD	Laboratory duplicate	A
S-3, S-4, S-5, S-6, S-7, S-16, S-20	A-2, A-3, A-4, A-5	Sensitivity/Contamination	Metal <QL	Method blank	A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Soil/Solids[1]
Analytical Group[2]	Asbestos
Concentration Level	TBD

Sampling Procedure[3]	Analytical Method SOP[4]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-3, S-4, S-5, S-6, S-17	A-8	Accuracy/Bias	Not determined	NA	NA
S-3, S-4, S-5, S-6, S-17	A-8	Precision	Not determined	Laboratory duplicate	A

Notes:

DQI	Data quality indicator
QL	Quantitation limit
%R	Percent recovery
RPD	Relative percent difference

1 Solids refer to building materials, soil invertebrates, or vegetation.
2 If information varies within an analytical group, separate by individual analyte.
3 Reference number from QAPP Worksheet #21
4 Reference number from QAPP Worksheet #23

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	VOA/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	Analytical Method SOP[3]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-12, S-13, S-15	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	VOC < QL	Trip blank	S & A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	VOC < QL	Rinsate blank	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias	1,1-Dichloroethene: 61-145 %R TCE: 71-120 %R Benzene: 76-127 %R Toluene: 76-125 %R Chlorobenzene: 75-130 %R	MS/MSD	S & A
S-12, S-13, S-15	A-1	Precision	1,1-Dichloroethene: 14% RPD TCE: 14% RPD Benzene: 11% RPD Toluene: 13% RPD Chlorobenzene: 13% RPD	MS/MSD	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	VOA/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	Analytical Method SOP[3]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-12, S-13, S-15	A-1	Accuracy	Vinyl chloride-d_3: 65-131 %R Chloroetahne-d_5: 71-131 %R 1,1-Dichloroethene-d_2: 55-104 %R 2-Butanone-d_5: 49-155 %R Chloroform-d: 78-121 %R 1,2-Dichloroethane-d_4: 78-129 %R Benzene-d_6: 77-124 %R 1,2-Dichloropropane-d_6 : 79-124 %R Toluene-d_8 : 77-121 %R 1,1,2,2-Tetrachloroethane-d_2: 73-125 %R Trans-1,3-Dichloropropene-d_4: 73-121 %R 2-Hexanone-d_5: 28-135 %R 1,4-Dioxane-d_8: 50-150 %R 1,2-Dichlorobnzene-d_4: 80-131 %R	Deuterated monitoring compounds	A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	VOC < QL	Method blank	A
S-12, S-13, S-15	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	SVOA/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-12, S-13, S-15	A-1	Precision	RPD ≤ 70%	Field Duplicate	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias-Contamination	SVOC < QL	Rinsate blank	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias	Phenol: 12-110 %R 2-Chlorophenol: 27-123 %R N-Nitroso-di-n-propylamine: 41-116 %R 4-Chloro-3-methylphenol: 23-97 %R Acenaphthene: 46-118 %R 4-Nitrophenol: 10-80 %R 2,4-Dinitrotoluene: 24-96 %R Pentachlorophenol: 9-103 %R Pyrene: 26-127 %R	MS/MSD	S & A
S-12, S-13, S-15	A-1	Precision	Phenol: 42% RPD 2-Chlorophenol: 40% RPD N-Nitroso-di-n-propylamine: 38% RPD 4-Chloro-3-methylphenol: 42% RPD Acenaphthene: 31% RPD 4-Nitrophenol: 50% RPD 2,4-Dinitrotoluene: 38% RPD Pentachlorophenol: 50% RPD Pyrene: 31% RPD	MS/MSD	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	SVOA/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	Analytical Method SOP[3]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
S-12, S-13, S-15	A-1	Accuracy	Phenol-d_5: 39-106 %R Bis(2-chloroethyl)ether-d_8: 40-105 %R 2-Chlorophenol-d_4: 41-106 %R 4-Methylphenol-d_8: 25-111 %R Nitrobenzene-d_5: 43-108 %R 2-Nitrophenol-d_4: 40-108 %R 2,4-Dichlorophenol-d_3: 37-105 %R 4-Chloroaniline-d_4: 1-145 %R Dimethylphthalate-d_6: 47-114 %R Acenaphthylene-d_8: 41-107 %R 4-Nitrophenol-d_4: 33-116 %R Fluorene-d_{10}: 42-111 %R 4,6-Dinitro-2-methylphenol-d_2: 22-104 %R Anthracene-d_{10}: 44-110 %R Pyrene-d_{10}: 52-119 %R Benzo(a)pyrene-d_{12}: 32-121 %R Fluoranthene-d_{10}: 50-150 %R 2-Methylnaphthalene-d_{10}: 50-150 %R	Deuterated monitoring compounds	A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	SVOC < QL	Method blank	A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	SVOA/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-12, S-13, S-15	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	PCB/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-12, S-13, S-15	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias-Contamination	PCB < QL	Rinsate blank	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias	Aroclor-1016: 29-135 %R Aroclor-1260: 29-135 %R	MS/MSD	S & A
S-12, S-13, S-15	A-1	Precision	Aroclor-1016: 15% RPD Aroclor-1260: 20% RPD	MS/MSD	S & A
S-12, S-13, S-15	A-1	Accuracy	Decachlorobiphenyl: 30-150 %R	Surrogate spike	A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	PCB < QL	Method blank	A
S-12, S-13, S-15	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	Pesticide/CLP
Concentration Level	Low concentration

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-12, S-13, S-15	A-1	Precision	RPD ≤ 70%	Field duplicate	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias-Contamination	Pesticide < QL	Rinsate blank	S & A
S-12, S-13, S-15	A-1	Accuracy/Bias	Gamma-BHC: 56-123 %R Heptachlor: 40-131 %R Aldrin: 40-120 %R Dieldrin : 52-126 %R Endrin : 56-121 %R 4,4'-DDT : 38-127 %R	MS/MSD	S & A
S-12, S-13, S-15	A-1	Precision	Gamma-BHC: 15% RPD Heptachlor: 20% RPD Aldrin: 22% RPD Dieldrin: 18% RPD Endrin: 21% RPD 4,4'-DDT: 27% RPD	MS/MSD	S & A
S-12, S-13, S-15	A-1	Accuracy	Tetrachloro-m-xylene: 30-150 %R	Surrogate spike	A
S-12, S-13, S-15	A-1	Accuracy/ Bias-Contamination	Pesticide < QL	Method blank	A
S-12, S-13, S-15	A-1	Completeness	≥ 90%	Data completeness defined as data not qualified as rejected after validation	S & A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	TAL Metals, Mercury, Cyanide /CLP
Concentration Level	Multi-concentration

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-12, S-13, S-15, S-21	A-2	Precision	RPD ≤ 70%	Field duplicate	S & A
S-12, S-13, S-15, S-21	A-2	Accuracy/Bias-Contamination	Metal < QL	Rinsate blank	S & A
S-12, S-13, S-15, S-21	A-2	Accuracy	All metals: 75-125 %R	MS	A
S-12, S-13, S-15, S-21	A-2	Precision	All metals: < 20% RPD	Laboratory duplicate	A
S-12, S-13, S-15, S-21	A-2	Sensitivity/Contamination	Metal < QL	Method blank	A

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Water
Analytical Group[1]	Hardness
Concentration Level	TBD

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-15	A-10	Accuracy/Bias	100.84 %R (Method Limit)	Rinsate blank	S & A
		Precision	4.2% RSD (Method Limit)	Laboratory duplicate	A

Notes:

DQI	Data quality indicator
QL	Quantitation limit
%R	Percent recovery
RPD	Relative percent difference
RSD	Relative standard deviation

1 If information varies within an analytical group, separate by individual analyte.
2 Reference number from QAPP Worksheet #21
3 Reference number from QAPP Worksheet #23

QAPP WORKSHEET #12 (CONTINUED)
MEASUREMENT PERFORMANCE CRITERIA TABLE

Matrix	Air
Analytical Group[1]	Asbestos
Concentration Level	TBD

Sampling Procedure[2]	**Analytical Method SOP**[3]	**DQIs**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)**
S-1, S-2	A-9	Accuracy/Bias	Not determined	NA	NA
S-1, S-2	A-9	Precision	Not determined	Laboratory duplicate	A

Notes:

DQI Data quality indicator

1 If information varies within an analytical group, separate by individual analyte.
2 Reference number from QAPP Worksheet #21
3 Reference number from QAPP Worksheet #23

QAPP WORKSHEET #13
SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

(UFP QAPP Section 2.7)
Identify all secondary data and information that will be used for the project and their originating sources. Specify how the secondary data will be used and the limitations on their use.

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Source (Originating Org, Data Types, data Generation/Collection Dates)	How data will be used	Limitation on Data Use
Soil, groundwater, and surface water sampling data	IEPA. "CERCLA Preliminary Assessment Report." Carus Chemical Company. September 1991.	IEPA; soil, groundwater, surface water samples; collected before 1991	OU2 data will be used as a starting point to characterize the nature of contamination at OU2, and any OU1 data will be compared to OU2 data.	None
Soil, groundwater, and surface water sampling data	IEPA. "CERCLA Screening Site Inspection Report." Carus Chemical Company. December 1992.	IEPA; soil, groundwater, surface water samples; collected before 1992	OU1 data apply to the shallow slag pile, and analytical results will be compared to OU2 data.	None
Soil, groundwater, and surface water sampling data	Geosyntec. "Preliminary Site Investigation Report." Carus Chemical Company, June 1993.	Geosyntec; soil, groundwater, surface water samples; collected before 1993	OU1 data apply to the shallow slag pile, and analytical results will be compared to OU2 data.	None
Soil, groundwater, and surface water sampling data	Geosyntec. "Phase I Site Investigation Completion Report." Carus Chemical Company Manufacturing Facility, LaSalle, Illinois. January 1994.	Geosyntec; soil, groundwater, surface water samples; collected before 1994	OU1 data apply to the shallow slag pile, and analytical results will be compared to OU2 data.	None
Soil, groundwater, and surface water sampling data	IEPA. "CERCLA Preliminary Assessment Report." M&H Zinc Company Site. October 1994.	IEPA; soil, groundwater, surface water samples; collected before 1994	OU2 data will be used as a starting point to characterize the nature of contamination at the site.	None

QAPP WORKSHEET #13 (CONTINUED)
SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Source (Originating Org, Data Types, data Generation/Collection Dates)	How data will be used	Limitation on Data Use
Soil, groundwater, and surface water sampling data	IEPA. "CERCLA Integrated Site Assessment Report." M&H Zinc Company Site. January 1995.	IEPA; soil, groundwater, surface water samples; collected before 1995	OU2 data will be used as a starting point to characterize the nature of contamination at the site.	None
Soil, groundwater, and surface water sampling data	Geosyntec. "Phase II Site Investigation Completion Report." Carus Chemical Company Manufacturing Facility, LaSalle, Illinois. January 1996.	Geosyntec; soil, groundwater, surface water samples; collected before 1996	OU1 data apply to the shallow slag pile, and analytical results will be compared to OU2 data.	None
Residential soil sampling data	IDPH. "Matthiessen Hegeler Zinc Co., LaSalle Residential Soil Sampling Results." January 2000.	IDPH; residential soil samples; collected before 2000	OU2 data will be used as a starting point to characterize the nature of contamination at the site.	None
Soil, groundwater, and surface water sampling data	Geosyntec. "Technical Letter Report Summary." M&H Zinc Company Site, LaSalle, Illinois. December 2006.	Geosyntec; soil, groundwater, surface water samples; collected before 2006	OU1 data apply to the shallow slag pile, and analytical results will be compared to OU2 data.	None
Soil, groundwater, and surface water sampling data	SulTRAC. "Data Evaluation Report OU2 Phase I." M&H Zinc Company Site, LaSalle, Illinois. February 2008.	SulTRAC; Phase I sample results for soil, groundwater, and surface water; collected in Summer/Fall 2007	OU2 data will be used to characterize the site and develop the Phase II planning documents, and for preliminary preparation of the RI report.	None
Soil, groundwater, and surface water sampling data	Geosyntec. "Data Evaluation Report Phase I Remedial Investigation Characterization Operable Unit 1." M&H Zinc Company Site, LaSalle, Illinois. April 2008	Geosyntec; Phase I sample results for soil, groundwater, and, surface water; collected in Fall/Winter 2007	OU1 data will be used to continue characterization and delineation of the OU1 site and for preliminary preparation of the RI report.	None

Note:
IDPH Illinois Department of Public Health

QAPP WORKSHEET #14
SUMMARY OF PROJECT TASKS

(UFP QAPP Section 2.8.1)
Provide a brief overview of the listed project activities.

Sampling Tasks:

1. Conduct asbestos air monitoring at two locations inside the rolling mill and at two locations in the former main industrial plant area where analytical results indicated the highest detections of asbestos in soils during Phase I. The highest asbestos concentrations were detected during Phase I inside the former main industrial plant.
2. Collect soil samples from 60 soil borings at two distinct depths (surface and subsurface).
3. Collect solid samples from 50 building structures.
4. Perform soil XRF screenings in remote areas not sampled using the Geoprobe®, and collect approximately 50 surface soil samples for CLP analysis.
5. Collect groundwater samples from 19 Phase I monitoring wells during June 2008 and then from the additional 17 newly installed Phase II monitoring wells (total of 36 monitoring wells) thereafter.
6. Collect surface water samples from eight locations at the beginning and end of Summer 2008.
7. Collect soil, sinter, and slag samples for bioassessibility testing.
8. Collect paired vegetation and soil invertebrate or soil, sinter, slag samples for bioavailability testing.
9. Take digital photographs to document activities.
10. Log activities and tasks in field notebook.
11. Prepare sample documentation such as chain-of-custody forms, sample labels, custody seals, etc.

Analysis Tasks: The CLP laboratory will analyze samples for VOCs, SVOCs, PCBs, pesticides, TAL metals (including mercury) and cyanide, TCLP metals, and SPLP metals. A subcontracted laboratory will analyze samples for asbestos in air samples, with performance evaluation samples (provided by the EPA SMO). SulTRAC also anticipates submitting soil/sinter/slag samples for bioassessibility to a subcontracted laboratory. Additionally, SulTRAC anticipates submitting paired vegetation and soil invertebrate samples to a subcontracted laboratory for tissue analyses, with a contingency plan of sending to a subcontracted laboratory representative soil, sinter, and slag samples in which to grow lettuce and to determine earthworm lethality.

QC Tasks: The following QC samples will be collected and analyzed during the sampling event: field duplicates, MS/MSD samples, rinsate blanks, and trip blanks.

Secondary Data: See Worksheet #13

QAPP WORKSHEET #14 (CONTINUED)
SUMMARY OF PROJECT TASKS

Data Management Tasks: Analytical data will be archived in an electronic database after validation.
Documentation and Records: All samples collected will be documented in a logbook using a ballpoint pen. The time of collection, identification number, sampling location, field observations, sampler's name, and analyses will be recorded in the logbook for each sample. Each page of the logbook will be dated, numbered, and signed by SulTRAC personnel. Field data records will be maintained at SulTRAC's Chicago office. SulTRAC will follow custody procedures outlined in SulTRAC's program-level QAPP for the RAC 2 contract. Further specifications are described in the FSP.
Assessment/Audit Tasks: An audit of one off-site laboratory (asbestos analysis) is planned as part of this project.
Data Review Tasks: EPA will perform limited CADRE for all CLP data and will prepare a case narrative detailing any issues or inconsistencies discovered. SulTRAC will review data generated by subcontracted laboratories. The SulTRAC project manager will review the case narrative and will detail any analytical issues that may potentially affect data quality in the RI/FS report.

QAPP WORKSHEET #15
REFERENCE LIMITS AND EVALUATION TABLE

(UFP QAPP Section 2.8.1)
Complete this worksheet for each matrix.
Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the QLs that must be met to achieve the PQOs. Finally, list the published and achievable detection and QLs for each analyte.

Reference Limits Table – Soil

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
VOA/CLP	Dichlorodifluoromethane	75-71-8	9.4E+01	5.0E-03
VOA/CLP	Chloromethane	74-87-3	4.7E+01	5.0E-03
VOA/CLP	Vinyl chloride	75-01-4	7.9E-02	5.0E-03
VOA/CLP	1,2,4-Trichlorobenzene	87-61-6	6.2E+01	5.0E-03
VOA/CLP	Trichlorofluoromethane	75-69-4	3.9E+02	5.0E-03
VOA/CLP	Bromomethane	75-27-2	3.9E+00	5.0E-03
VOA/CLP	Chloroethane	75-00-3	3.0E+00	5.0E-03
VOA/CLP	Trichlorofluoromethane	75-69-4	3.9E+02	5.0E-03
VOA/CLP	1,1-Dichloroethene	75-35-4	1.2E+02	5.0E-03
VOA/CLP	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NC	5.0E-03
VOA/CLP	Acetone	67-64-1	1.4E+04	1.0E-02
VOA/CLP	Carbon disulfide	75-15-0	3.6E+02	5.0E-03
VOA/CLP	Methyl acetate	79-20-9	2.2E+04	5.0E-03
VOA/CLP	Methylene chloride	75-09-2	9.1E+00	5.0E-03
VOA/CLP	Trans-1,2-Dichloroethene	156-50-5	6.9E+01	5.0E-03
VOA/CLP	Methyl tert-butyl ether	1634-04-4	3.2E+01	5.0E-03
VOA/CLP	1,1-Dichloroethane	75-34-3	5.1E+02	5.0E-03
VOA/CLP	Cis-1,2-Dichloroethene	540-59-0	4.3E+01	5.0E-03
VOA/CLP	2-Butanone	78-93-3	2.2E+04	1.0E-02
VOA/CLP	Bromochloroform	74-97-5	NC	5.0E-03
VOA/CLP	Chloroform	67-66-3	2.2E-01	5.0E-03
VOA/CLP	1,1,1-Trichloroethane	71-55-6	1.2E+03	5.0E-03

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
VOA/CLP	Cyclohexane	110-82-7	1.4E+02	5.0E-03
VOA/CLP	Carbon tetrachloride	56-23-5	2.5E-01	5.0E-03
VOA/CLP	Benzene	71-43-2	6.4E-01	5.0E-03
VOA/CLP	1,2-Dichloroethane	67-66-3	2.8E-01	5.0E-03
VOA/CLP	1,4-Dioxane	123-91-1	4.4E+01	1.0E-01
VOA/CLP	TCE	79-01-6	5.3E-02	5.0E-03
VOA/CLP	Methylcyclohexane	108-87-2	2.6E+03	5.0E-03
VOA/CLP	1,2-Dichloropropane	78-87-5	3.4E-01	5.0E-03
VOA/CLP	Bromodichloromethane	75-27-4	8.2E-01	5.0E-03
VOA/CLP	Cis-1,3-Dichloropropene	26952-23-8	7.8E-01	5.0E-03
VOA/CLP	4-Methyl-2-pentanone	108-10-1	5.3E+03	1.0E-02
VOA/CLP	Toluene	108-88-3	5.2E+02	5.0E-03
VOA/CLP	Trans-1,3-Dichloropropene	10061-02-6	NC	5.0E-03
VOA/CLP	1,1,2-Trichloroethane	79-00-5	7.3E-01	5.0E-03
VOA/CLP	Tetrachloroethene	127-18-4	4.8E-01	5.0E-03
VOA/CLP	2-Henanone	591-78-6	NC	1.0E-02
VOA/CLP	Dibromochloromethane	75-25-2	1.1E+00	5.0E-03
VOA/CLP	1,2-Dibromoethane	106-93-4	3.2E-02	5.0E-03
VOA/CLP	Chlorobenzene	108-90-7	1.5E+02	5.0E-03
VOA/CLP	Ethylbenzene	100-41-4	4.0E+02	5.0E-03
VOA/CLP	o-Xylene	1330-20-7	2.7E+02	5.0E-03
VOA/CLP	m,p-Xylene	1330-20-7	2.7E+02	5.0E-03
VOA/CLP	Styrene	100-42-5	1.7E+03	5.0E-03
VOA/CLP	Bromoform	75-25-2	6.2E+01	5.0E-03
VOA/CLP	Isopropylbenzene	98-82-8	NC	5.0E-03
VOA/CLP	1,1,2,2-Tetrachloroethane	79-34-5	4.1E-01	5.0E-03
VOA/CLP	1,3-Dichlorobenzene	95-50-1	5.3E+02	5.0E-03
VOA/CLP	1,4-Dichlorobenzene	95-50-1	3.4E+00	5.0E-03
VOA/CLP	1,2-Dichlorobenzene	95-50-1	6.0E+02	5.0E-03
VOA/CLP	1,2-Dibromo-3-chloropropane	96-12-8	4.6E-01	5.0E-03
VOA/CLP	1,2,4-Trichlorobenzene	120-82-1	6.2E+01	5.0E-03
VOA/CLP	1,2,3-Trichlorobenzene	87-61-6	NC	5.0E-03

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
SVOA/CLP	Phenol	108-95-2	1.8E+04	1.7E-01
SVOA/CLP	Bis (2-Chloroethyl) ether	111-44-4	2.2E-01	1.7E-01
SVOA/CLP	2-Chlorophenol	95-57-8	6.3E+01	1.7E-01
SVOA/CLP	2-Methylphenol	95-48-7	3.1E+03	1.7E-01
SVOA/CLP	2,2'-Oxybis(1-chloropropane)	108-60-1	NC	1.7E-01
SVOA/CLP	Acetophenone	98-86-2	NC	1.7E-01
SVOA/CLP	4-Methylphenol	106-44-5	3.1E+02	1.7E-01
SVOA/CLP	N-Nitroso-di-n propylamine	621-64-7	6.9E-02	1.7E-01
SVOA/CLP	Hexachloroethane	67-72-1	3.5E+01	1.7E-01
SVOA/CLP	Nitrobenzene	98-95-3	2.0E+01	1.7E-01
SVOA/CLP	Isophorone	78-59-1	5.1E+02	1.7E-01
SVOA/CLP	2-Nitrophenol	88-75-5	NC	1.7E-01
SVOA/CLP	2,4-Dimethylphenol	105-67-9	1.2E+03	1.7E-01
SVOA/CLP	Bis(2-Chloroethoxy) methane	111-91-1	NC	1.7E-01
SVOA/CLP	2,4-Dichlorophenol	120-83-2	1.8E+02	1.7E-01
SVOA/CLP	Naphthalene	91-20-3	5.6E+01	1.7E-01
SVOA/CLP	4-Chloroaniline	106-47-8	2.4E+02	1.7E-01
SVOA/CLP	Hexachlorobutadiene	87-68-3	6.2E+00	1.7E-01
SVOA/CLP	Caprolactam	105-60-2	3.1E+04	1.7E-01
SVOA/CLP	4-Chloro-3-methylphenol	59-50-7	NC	1.7E-01
SVOA/CLP	2-Methylnaphthalene	91-57-6	NC	1.7E-01
SVOA/CLP	Hexachlorocyclopentadiene	77-47-4	3.7E+02	1.7E-01
SVOA/CLP	2,4,6-Trichlorophenol	88-06-2	6.1E+00	1.7E-01
SVOA/CLP	2,4,5-Trichlorophenol	95-95-4	6.1E+03	1.7E-01
SVOA/CLP	1,1-Biphenyl	92-52-4	3.0E+03	1.7E-01
SVOA/CLP	2-Chloronaphthalene	91-58-7	4.9E+03	1.7E-01
SVOA/CLP	2-Nitroaniline	88-74-4	1.8E+02	3.30E-01
SVOA/CLP	Dimethylphthalate	131-11-3	1.0E+05	1.7E-01
SVOA/CLP	2,6-Dinitrotoluene	606-20-2	6.1E+01	1.7E-01
SVOA/CLP	Acenaphthylene	208-96-8	37.0E+02	1.7E-01
SVOA/CLP	3-Nitroaniline	99-09-2	1.8E+01	3.30E-01
SVOA/CLP	Acenaphthene	83-32-9	3.7E+03	1.7E-01
SVOA/CLP	2,4-Dinitrophenol	51-28-5	1.2E+02	3.30E-01

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
SVOA/CLP	Dibenzofuran	132-64-9	1.5E+02	1.7E-01
SVOA/CLP	2,4-Dinitrotoluene	121-14-2	1.2E+02	1.7E-01
SVOA/CLP	Diethylphthalate	84-66-2	4.9E+04	1.7E-01
SVOA/CLP	Fluorene	86-73-7	27.0E+02	1.7E-01
SVOA/CLP	4-Chlorophenyl-phenyl ether	7005-72-3	NC	1.7E-01
SVOA/CLP	4-Nitroaniline	100-01-6	2.3E+01	3.30E-01
SVOA/CLP	2-Methyl-4,6-dinitro phenol	534-52-1	6.1E+00	3.30E-01
SVOA/CLP	N-Nitrosodiphenylamine	86-30-6	9.9E+01	1.7E-01
SVOA/CLP	1,2,4,5-Tetrachlorobenzene	95-94-3	1.8E+01	1.7E-01
SVOA/CLP	4-Bromophenyl-phenylether	101-55-3	NC	1.7E-01
SVOA/CLP	Hexachlorobenzene	118-74-1	3.0E-01	1.7E-01
SVOA/CLP	Atrazine	1912-24-9	2.2E+00	1.7E-01
SVOA/CLP	Pentachlorophenol	87-86-5	3.0E+00	3.30E-01
SVOA/CLP	Phenanthrene	85-014-8	22E+03	1.7E-01
SVOA/CLP	Anthracene	20-12-7	22E+03	1.7E-01
SVOA/CLP	Carbazole	86-74-8	2.4E+01	1.7E-01
SVOA/CLP	Di-n-butylphthalate	84-74-2	6.1E+03	1.7E-01
SVOA/CLP	Fluoranthene	206-44-0	23E+02	1.7E-01
SVOA/CLP	Pyrene	129-00-0	2.3E+03	1.7E-01
SVOA/CLP	Butylbenzylphthalate	85-68-7	1.2E+04	1.7E-01
SVOA/CLP	3,3'-dichlorobenzidine	91-94-1	1.1E+00	1.7E-01
SVOA/CLP	Benzo(a)anthracene	56-55-3	0.62E+00	1.7E-01
SVOA/CLP	Benzo(b)fluoranthene	205-99-2	6.2E-01	1.7E-01
SVOA/CLP	Benzo(k)fluoranthene	207-08-9	6.2E+00	1.7E-01
SVOA/CLP	Chrysene	218-01-9	3.8E+00	1.7E-01
SVOA/CLP	Bis(2-ethylhexyl)phthalate	117-81-7	3.5E+01	1.7E-01
SVOA/CLP	Di-n-octylphthalate	117-84-0	2.4E+03	1.7E-01
SVOA/CLP	Benzo(a) pyrene	50-32-8	6.2E-02	1.7E-01
SVOA/CLP	Indeno(1,2,3,-cd)pyrene	193-39-5	6.2E-01	1.7E-01
SVOA/CLP	Dibenzo(a,h)anthracene	53-70-3	6.2E-02	1.7E-01
SVOA/CLP	Benzo(g,h,i) perylene	191-24-2	NC	1.7E-01

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
SVOA/CLP	2,3,4,6-Tetrachlorophenol	58-90-2	1.8E+03	1.7E-01
PCBs/CLP	Aroclor-1016[2]	12674-11-2	3.9E+00	3.3E-02
PCBs/CLP	Aroclor-1221	11104-28-2	NC	3.3E-02
PCBs/CLP	Aroclor-1232	11141-16-5	NC	3.3E-02
PCBs/CLP	Aroclor-1242	53469-21-9	NC	3.3E-02
PCBs/CLP	Aroclor-1248	12672-29-6	NC	3.3E-02
PCBs/CLP	Aroclor-1254[3]	11097-69-1	2.2E-01	3.3E-02
PCBs/CLP	Aroclor-1260	11096-82-5	NC	3.3E-02
PCBs/CLP	Aroclor-1268	11100-14-4	NC	3.3E-02
Pesticides/CLP	alpha-BHC	608-73-1	9.0E-02	1.7E-03
Pesticides/CLP	Beta-BHC	608-73-1	3.2E-01	1.7E-03
Pesticides/CLP	delta-BHC	608-73-1	3.2E-01	1.7E-03
Pesticides/CLP	gamma-BHC (Lindane)	608-73-1	4.4E-01	1.7E-03
Pesticides/CLP	Heptachlor	76-44-8	1.1E-01	1.7E-03
Pesticides/CLP	Aldrin	309-00-2	2.9E-02	1.7E-03
Pesticides/CLP	Heptachlor epoxide	1024-57-3	5.3E-02	1.7E-03
Pesticides/CLP	Endosulfan I	115-29-7	3.7E+02	1.7E-03
Pesticides/CLP	Dieldrin	60-57-1	3.0E-02	3.3E-03
Pesticides/CLP	4,4'-DDE	72-55-9	1.7E+00	3.3E-03
Pesticides/CLP	Endrin	72-20-8	1.8E+01	3.3E-03
Pesticides/CLP	Endosulfan II	115-29-7	3.7E+02	3.3E-03
Pesticides/CLP	4,4'-DDD	72-54-8	2.4E+00	3.3E-03
Pesticides/CLP	4,4'-DDT	50-29-3	1.7E+00	3.3E-03
Pesticides/CLP	Endosulfan sulfate	1031-07-8	NC	3.3E-03
Pesticides/CLP	Methoxychlor	72-43-5	3.1E+02	1.7E-02
Pesticides/CLP	Endrin ketone	72-20-8	1.8E+01	3.3E-03
Pesticides/CLP	Endrin aldehyde	72-20-8	1.8E+01	3.3E-03
Pesticides/CLP	alpha-Chlordane	57-74-9	1.6E+00	1.7E-03
Pesticides/CLP	gamma-Chlordane	57-74-9	1.6E+00	1.7E-03

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit - Soil (mg/kg)[1]	CRQL - Soil (mg/kg)
Pesticides/CLP	Toxaphene	8001-35-2	4.4E-01	1.7E-01
TAL Metals/CLP	Aluminum	7429-90-5	7.6E+04	20.0E+00
TAL Metals/CLP	Antimony	7440-36-0	3.1E+01	6.0E+00
TAL Metals/CLP	Arsenic[4]	7440-38-2	11.3E+00	1.0E+00
TAL Metals/CLP	Barium	7440-39-3	5.4E+03	20.0E+00
TAL Metals/CLP	Beryllium	7440-41-7	1.5E+02	0.5E+00
TAL Metals/CLP	Cadmium	7440-43-9	3.7E+01	0.5E+00
TAL Metals/CLP	Calcium	17852-99-2	NC	500.0E+00
TAL Metals/CLP	Chromium	7440-47-3	1.0E+05	1.0E+00
TAL Metals/CLP	Cobalt	7440-48-4	9.0E+02	5.0E+00
TAL Metals/CLP	Copper	7440-50-8	3.1E+03	2.5E+00
TAL Metals/CLP	Iron	7439-89-6	2.3E+04	10.0E+00
TAL Metals/CLP	Lead	7439-92-1	4.0E+02	1.0E+00
TAL Metals/CLP	Magnesium	7439-95-4	NC	500.0E+00
TAL Metals/CLP	Manganese	7439-96-5	1.8E+03	1.5E+00
TAL Metals/CLP	Mercury	7439-97-6	2.3E+01	0.1E+00
TAL Metals/CLP	Nickel	7440-02-0	1.6E+02	4.0E+00
TAL Metals/CLP	Potassium	7440-22-4	NC	500.0E+00
TAL Metals/CLP	Selenium	7782-49-2	3.9E+02	3.5E+00
TAL Metals/CLP	Silver	7440-22-4	3.9E+02	1.0E+00
TAL Metals/CLP	Sodium	7440-23-5	NC	500.0E+00
TAL Metals/CLP	Thallium	7440-28-0	5.2E+00	2.5E+00
TAL Metals/CLP	Vanadium	7440-62-2	7.8E+01	5.0E+00
TAL Metals/CLP	Zinc	7440-66-6	2.3E+04	6.0E+00
TAL Cyanide/CLP	Cyanide	74-90-8	1.2E+03	2.5E+00
Asbestos	Asbestos	NC	NA	NA

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Notes:

CAS	Chemical Abstract Services
CRQL	Contract-required quantitation limit
mg/kg	Milligram per kilogram
NC	No criteria
NA	Not available

1	Region 9 residential preliminary remediation goal (PRG), October 2004
2	PCBs (unspeciated mixture, low risk; for example, Aroclor 1016)
3	PCBs (unspeciated mixture, high risk; for example, Aroclor 1254)
4	11.3 mg/kg is the background value for arsenic (Title 35 of the *Illinois Administrative Code*, Part 742, Appendix A, Table G)

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Reference Limits Table –Water

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
VOA/CLP	Dichlorodifluoromethane	75-71-8	3.9E+02	5.0E+00
VOA/CLP	Chloromethane	74-87-3	1.6E+02	5.0E+00
VOA/CLP	Vinyl chloride	75-01-4	2.0E-02	5.0E+00
VOA/CLP	Bromomethane	75-27-2	8.7E+00	5.0E+00
VOA/CLP	Chloroethane	75-00-3	4.6E+00	5.0E+00
VOA/CLP	Trichlorofluoromethane	75-69-4	1.3E+03	5.0E+00
VOA/CLP	1,1-Dichloroethene	75-35-4	3.4E+02	5.0E+00
VOA/CLP	Acetone	67-64-1	5.5E+03	10.0E+00
VOA/CLP	Carbon disulfide	75-15-0	1.0E+03	5.0E+00
VOA/CLP	Methyl acetate	79-20-9	6.1E+03	5.0E+00
VOA/CLP	Methylene chloride	75-09-2	4.3E+00	5.0E+00
VOA/CLP	Trans-1,2-Dichloroethene	156-50-5	1.2E+02	5.0E+00
VOA/CLP	Methyl tert-butyl ether	1634-04-4	1.1E+01	5.0E+00
VOA/CLP	1,1-Dichloroethane	75-34-3	8.1E+02	5.0E+00
VOA/CLP	Cis-1,2-Dichloroethene	540-59-0	6.1E+01	5.0E+00
VOA/CLP	2-Butanone	78-93-3	7.0E+03	10.0E+00
VOA/CLP	Bromochloroform	74-97-5	NC	5.0E+00
VOA/CLP	Chloroform	67-66-3	1.7E-01	5.0E+00
VOA/CLP	1,1,1-Trichloroethane	71-55-6	3.2E+03	5.0E+00
VOA/CLP	Cyclohexane	110-82-7	1.0E+04	5.0E+00
VOA/CLP	Carbon tetrachloride	56-23-5	1.7E-01	5.0E+00
VOA/CLP	Benzene	71-43-2	3.5E-01	5.0E+00
VOA/CLP	1,2-Dichloroethane	67-66-3	1.2E-01	5.0E+00
VOA/CLP	1,4-Dioxane	123-91-1	6.1E+00	1.0E+02
VOA/CLP	TCE	79-01-6	NC	5.0E+00
VOA/CLP	Methylcyclohexane	108-87-2	5.2E+03	5.0E+00
VOA/CLP	1,2-Dichloropropane	78-87-5	1.6E-01	5.0E+00
VOA/CLP	Bromodichloromethane	75-27-4	1.8E-01	5.0E+00

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
VOA/CLP	Cis-1,3-Dichloropropene	26952-23-8	4.0E-01	5.0E+00
VOA/CLP	4-Methyl-2-pentanone	108-10-1	2.0E+03	1.0E+01
VOA/CLP	Toluene	108-88-3	7.2E+02	5.0E+00
VOA/CLP	Trans-1,3-Dichloropropene	10061-02-6	NC	5.0E+00
VOA/CLP	1,1,2-Trichloroethane	79-00-5	2.0E-01	5.0E+00
VOA/CLP	Tetrachloroethene	127-18-4	1.0E-01	5.0E+00
VOA/CLP	2-Hexanone	591-78-6	NC	1.0E+01
VOA/CLP	Dibromochloromethane	75-25-2	1.3E-01	5.0E+00
VOA/CLP	1,2-Dibromoethane	106-93-4	5.6E-03	5.0E+00
VOA/CLP	Chlorobenzene	108-90-7	1.1E+02	5.0E+00
VOA/CLP	Ethylbenzene	100-41-4	1.3E+03	5.0E+00
VOA/CLP	o-Xylene	1330-20-7	2.1E+02	5.0E+00
VOA/CLP	m,p-Xylene	1330-20-7	2.1E+02	5.0E+00
VOA/CLP	Styrene	100-42-5	1.6E+03	5.0E+00
VOA/CLP	Bromoform	75-25-2	8.5E+00	5.0E+00
VOA/CLP	1,1,2,2-Tetrachloroethane	79-34-5	5.5E-02	5.0E+00
VOA/CLP	1,3-Dichlorobenzene	95-50-1	1.8E+02	5.0E+00
VOA/CLP	1,4-Dichlorobenzene	95-50-1	5.0E-01	5.0E+00
VOA/CLP	1,2-Dichlorobenzene	95-50-1	3.7E+02	5.0E+00
VOA/CLP	1,2-Dibromo-3-chloropropane	96-12-8	4.8E-02	5.0E+00
VOA/CLP	1,2,4-Trichlorobenzene	120-82-1	7.2E+00	5.0E+00
VOA/CLP	1,2,3-Trichlorobenzene	87-61-6	NC	5.0E+00

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
SVOA/CLP	Benzaldehyde	100-52-7	3.6E+03	5.0E+00
SVOA/CLP	Phenol	108-95-2	1.1E+04	5.0E+00
SVOA/CLP	Bis (2-chloroethyl) ether	111-44-4	1.0E-02	5.0E+00
SVOA/CLP	2-Chlorophenol	95-57-8	3.0E+01	5.0E+00
SVOA/CLP	2-Methylphenol	95-48-7	1.8E+03	5.0E+00
SVOA/CLP	2,2'-Oxybis(1-chloropropane)	108-60-1	NC	5.0E+00
SVOA/CLP	4-Methylphenol	106-44-5	1.8E+02	5.0E+00
SVOA/CLP	Acetophenone	98-86-2	NC	5.0E+00
SVOA/CLP	N-Nitroso-di-n propylamine	621-64-7	9.6E-03	5.0E+00
SVOA/CLP	Hexachloroethane	67-72-1	4.8E+00	5.0E+00
SVOA/CLP	Nitrobenzene	98-95-3	3.4E+00	5.0E+00
SVOA/CLP	Isophorone	78-59-1	7.1E+01	5.0E+00
SVOA/CLP	2-Nitrophenol	88-75-5	NC	5.0E+00
SVOA/CLP	2,4-Dimethylphenol	105-67-9	7.3E+02	5.0E+00
SVOA/CLP	2,4-Dichlorophenol	120-83-2	1.1E+02	5.0E+00
SVOA/CLP	Naphthalene	91-20-3	6.2E+00	5.0E+00
SVOA/CLP	4-Chloroaniline	106-47-8	1.5E+02	5.0E+00
SVOA/CLP	Hexachlorobutadiene	87-68-3	8.6E-01	5.0E+00
SVOA/CLP	Caprolactam	105-60-2	1.8E+04	5.0E+00
SVOA/CLP	4-Chloro-3-methylphenol	59-50-7	NC	5.0E+00
SVOA/CLP	2-Methylnaphthalene	91-57-6	NC	5.0E+00
SVOA/CLP	Hexachlorocyclopentadiene	77-47-4	2.2E+02	5.0E+00
SVOA/CLP	2,4,6-Trichlorophenol	88-06-2	3.6E+00	5.0E+00
SVOA/CLP	2,4,5-Trichlorophenol	95-95-4	3.6E+03	5.0E+00
SVOA/CLP	1,1-Biphenyl	92-52-4	3.0E+02	5.0E+00
SVOA/CLP	2-Chloronaphthalene	91-58-7	4.9E+02	5.0E+00
SVOA/CLP	2-Nitroaniline	88-74-4	1.1E+02	1.0E+01
SVOA/CLP	4,6-Dinitro-2-methylphenol	534-52-1	3.6E+00	1.0E+01

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
SVOA/CLP	N-Nitrosodiphenylamine	86-30-6	1.4E+01	5.0E+00
SVOA/CLP	1,2,4,5-Tetrachlorobenzene	95-94-3	1.1E+01	5.0E+00
SVOA/CLP	Hexachlorobenzene	118-74-1	4.2E-02	5.0E+00
SVOA/CLP	2,4,6-Trichlorophenol	88-06-2	3.6E+00	5.0E+00
SVOA/CLP	2,4,5-Trichlorophenol	95-95-4	3.6E+03	5.0E+00
SVOA/CLP	Dimethylphthalate	131-11-3	3.6E+05	5.0E+00
SVOA/CLP	2,6-Dinitrotoluene	606-20-2	3.6E+01	5.0E+00
SVOA/CLP	Acenaphthylene	208-96-8	NC	5.0E+00
SVOA/CLP	3-Nitroaniline	99-09-2	3.2E+00	1.0E+01
SVOA/CLP	Acenaphthene	83-32-9	3.7E+02	5.0E+00
SVOA/CLP	2,4-Dinitrophenol	51-28-5	7.3E+01	1.0E+01
SVOA/CLP	4-Nitrophenol	100-02-7	NC	1.0E+01
SVOA/CLP	Dibenzofuran	132-64-9	1.2E+01	5.0E+00
SVOA/CLP	2,4-Dinitrotoluene	121-14-2	7.3E+01	5.0E+00
SVOA/CLP	Diethylphthalate	84-66-2	2.9E+04	5.0E+00
SVOA/CLP	Fluorene	86-73-7	2.4E+02	5.0E+00
SVOA/CLP	4-Chlorophenyl-phenyl ether	7005-72-3	NC	5.0E+00
SVOA/CLP	4-Nitroaniline	100-01-6	3.2E+00	1.0E+01
SVOA/CLP	Hexachlorobenzene	118-74-1	4.2E-02	5.0E+00
SVOA/CLP	Atrazine	1912-24-9	3.0E-01	5.0E+00
SVOA/CLP	Pentachlorophenol	87-86-5	5.6E-01	1.0E+01
SVOA/CLP	Phenanthrene	85-014-8	NC	5.0E+00
SVOA/CLP	Anthracene	20-12-7	1.8E+03	5.0E+00
SVOA/CLP	Carbazole	86-74-8	3.4E+00	5.0E+00
SVOA/CLP	Di-n-butylphthalate	84-74-2	3.6E+03	5.0E+00
SVOA/CLP	Fluoranthene	206-44-0	1.5E+03	5.0E+00

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
SVOA/CLP	Pyrene	129-00-0	1.8E+02	5.0E+00
SVOA/CLP	Butylbenzylphthalate	85-68-7	7.3E+03	5.0E+00
SVOA/CLP	3,3'-Dichlorobenzidine	91-94-1	1.5E-01	5.0E+00
SVOA/CLP	Benzo(a)anthracene	56-55-3	9.2E-02	5.0E+00
SVOA/CLP	Chrysene	218-01-9	9.2E+00	5.0E+00
SVOA/CLP	Bis (2-ethylhexyl) phthalate	117-81-7	4.8E+00	5.0E+00
SVOA/CLP	Di-n-octylphthalate	117-84-0	1.5E+03	5.0E+00
SVOA/CLP	Benzo(b)fluoranthene	205-99-2	9.2E-02	5.0E+00
SVOA/CLP	Benzo(k)fluoranthene	207-08-9	9.1E-02	5.0E+00
SVOA/CLP	Benzo(a)pyrene	50-32-8	9.3E-03	5.0E+00
SVOA/CLP	Indeno(1,2,3,-cd) pyrene	193-39-5	9.2E-02	5.0E+00
SVOA/CLP	Dibenzo(a,h) anthracene	53-70-3	9.2E-03	5.0E+00
SVOA/CLP	2,3,4,6-Tetrachlorophenol	58-90-03	1.1E+03	5.0E+00
PCB/CLP	Aroclor-1016[2]	12674-11-2	9.6E-01	1.0E+00
PCB/CLP	Aroclor-1221	11104-28-2	NC	1.0E+00
PCB/CLP	Aroclor-1232	11141-16-5	NC	1.0E+00
PCB/CLP	Aroclor-1242	53469-21-9	NC	1.0E+00
PCB/CLP	Aroclor-1248	12672-29-6	NC	1.0E+00
PCB/CLP	Aroclor-1254[3]	11097-69-1	3.4E-02	1.0E+00
PCB/CLP	Aroclor-1260	11096-82-5	NC	1.0E+00
PCB/CLP	Aroclor-1268	11100-14-4	NC	1.0E+00

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
Pesticide/CLP	alpha-BHC	608-73-1	NC	5.0E-02
Pesticide/CLP	beta-BHC	608-73-1	NC	5.0E-02
Pesticide/CLP	delta-BHC	608-73-1	NC	5.0E-02
Pesticide/CLP	gamma-BHC (Lindane)	608-73-1	NC	5.0E-02
Pesticide/CLP	Heptachlor	76-44-8	1.5E-02	5.0E-02
Pesticide/CLP	Aldrin	309-00-2	4.0E-03	5.0E-02
Pesticide/CLP	Heptachlor epoxide	1024-57-3	7.4E-03	5.0E-02
Pesticide/CLP	Endosulfan	115-29-7	2.2E+02	5.0E-02
Pesticide/CLP	Dieldrin	60-57-1	4.2E-03	1.0E-01
Pesticide/CLP	4,4'-DDE	72-55-9	NC	1.0E-01
Pesticide/CLP	Endrin	72-20-8	1.1E+01	1.0E-01
Pesticide/CLP	Endosulfan II	115-29-7	2.2E+02	1.0E-01
Pesticide/CLP	4,4'-DDD	72-54-8	NC	1.0E-01
Pesticide/CLP	Endosulfan sulfate	115-29-7	NC	1.0E-01
Pesticide/CLP	4,4'-DDT	50-29-3	NC	1.0E-01
Pesticide/CLP	Methoxychlor	72-43-5	NC	5.0E-01
Pesticide/CLP	Endrin ketone	72-20-8	NC	1.0E-01
Pesticide/CLP	Endrin aldehyde	72-20-8	NC	1.0E-01
Pesticide/CLP	alpha-Chlordane	57-74-9	1.9E-01	5.0E-02
Pesticide/CLP	gamma-Chlordane	57-74-9	1.9E-01	5.0E-02
Pesticide/CLP	Toxaphene	8001-35-2	6.1E-02	5.0E+00

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Analytical Group	Analyte	CAS Number	Project Action Limit – Water (µg/L)[1]	CRQL - Water (µg/L)
TAL Metals/CLP	Aluminum	7429-90-5	3.6E+04	2.0E+02
TAL Metals/CLP	Antimony	7440-36-0	1.5E+01	6.0E+01
TAL Metals/CLP	Arsenic	7440-38-2	4.5E-02	1.0E+01
TAL Metals/CLP	Barium	7440-39-3	2.6E+03	2.0E+02
TAL Metals/CLP	Beryllium	7440-41-7	7.3E+01	5.0E+00
TAL Metals/CLP	Cadmium	7440-43-9	1.8E+01	5.0E+00
TAL Metals/CLP	Calcium	17852-99-2	NC	5.0E+03
TAL Metals/CLP	Chromium	7440-47-3	5.5E+04	1.0E+01
TAL Metals/CLP	Cobalt	7440-48-4	7.3E+02	5.0E+01
TAL Metals/CLP	Copper	7440-50-8	1.5E+03	2.5E+01
TAL Metals/CLP	Iron	7439-89-6	1.1E+04	1.0E+02
TAL Metals/CLP	Lead[4]	7439-92-1	1.5E+01	1.0E+01
TAL Metals/CLP	Magnesium	7439-95-4	NC	5.0E+03
TAL Metals/CLP	Manganese	7439-96-5	8.8E+02	1.5E+01
TAL Metals/CLP	Mercury	7439-97-6	1.1E+01	2.0E-01
TAL Metals/CLP	Nickel	7440-02-0	7.3E+02	4.0E+01
TAL Metals/CLP	Potassium	7440-22-4	NC	5.0E+03
TAL Metals/CLP	Selenium	7782-49-2	1.8E+02	3.5E+01
TAL Metals/CLP	Silver	7440-22-4	1.8E+02	1.0E+01
TAL Metals/CLP	Sodium	7440-23-5	NC	5.0E+03
TAL Metals/CLP	Thallium	7440-28-0	2.4E+00	2.5E+01
TAL Metals/CLP	Vanadium	7440-62-2	3.6E+01	5.0E+01
TAL Metals/CLP	Zinc	7440-66-6	1.1E+04	6.0E+01
TAL Cyanide/CLP	Cyanide	74-90-8	7.3E+02	1.0E+01
Total Hardness	Total Hardness $CaCO_3$	NC	NC	NC

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Notes:

µg/L	Microgram per liter
CAS	Chemical Abstract Services
$CaCO_3$	Calcium carbonate
CRQL	Contract-required quantitation limit
NC	No criteria

1	Region 9 residential preliminary remediation goal (PRG), October 2004
2	PCBs (unspeciated mixture, low risk; for example, Aroclor 1016)
3	PCBs (unspeciated mixture, high risk; for example, Aroclor 1254)
4	Maximum contaminant level (MCL) from EPA 2006

QAPP WORKSHEET #16
PROJECT SCHEDULE/TIMELINE TABLE

(UFP QAPP Section 2.8.2)
List all project activities as well as the QA assessments that will be performed during the course of the project. Include the anticipated start and completion dates.

Activity	Organization	Date		Deliverable	Deliverable Due Date
		Anticipated Date of Initiation	Anticipated Date of Completion		
Phase II Field sampling	SulTRAC	July 2008	November/December 2008	Phase II FSP Phase II QAPP	30 days after Phase II work plan approval
Phase II Data evaluation	SulTRAC	December 2008	January/February 2009	Phase II Data Evaluation Summary Report	45 days after receipt of Phase II validated data
HHRA	SulTRAC	January 2009	March 2009	HHRA Report	Draft -45 days after receipt of Phase II Data Evaluation Summary Report Final -21 days after receipt of comments
ERA	SulTRAC	January 2009	March 2009	ERA Report	Draft -45 days after receipt of Phase II Data Evaluation Summary Report Final -21 days after receipt of comments
RI Report	SulTRAC/Geosyntec	March 2009	May/June2009	RI Report	Draft – 30 days after completion of HHRA or ERA which ever is later Final – 21 days after receipt of comments
OU2 Remedial Alternatives Screening	SulTRAC	May 2009	June/July 2009	Remedial Alternatives Screening Report	21 days after completion of RI Report

QAPP WORKSHEET #15 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE

Activity	Organization	Anticipated Date of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Remedial Alternatives Evaluation	SulTRAC	May 2009	June/July 2009	Remedial Alternatives Screening Report	21 days after completion of RI Report
Feasibility Study	SulTRAC/Geosyntec	June/July 2009	November 2009	Feasibility Study	Draft -45 days after completion of final RI Final -21 days after receipt of comments

QAPP WORKSHEET #17
SAMPLING DESIGN AND RATIONALE

(UFP QAPP Section 3.1.1)
Describe the project sampling approach. Provide the rationale for selecting sample locations and matrices for each analytical group and concentration level.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be collected, and the sampling frequency (including seasonal considerations). (May refer to map or Worksheet #18 for details).

Based on the preliminary results of the 2007 Phase I field investigation, the nature of contamination at the OU2 M&H Site has been fairly well characterized. The Phase II field investigation will further characterize the contamination sources and delineate the extent of contamination. Therefore, soil, solids, groundwater, and surface water samples will be collected as summarized below.

Prior to field activities, asbestos air samples will be collected from two locations inside the rolling mill during an active business day within the facility and two downwind locations from areas found to have the highest asbestos concentrations in surface soils within the main industrial area. SulTRAC anticipates submitting four air samples and approximately 30 soil, 50 building material, and 10 performance evaluation samples for asbestos analysis (provided by EPA SMO).

Surface and subsurface soil samples will be collected from 60 borings at two depth intervals per boring. The 60 soil borings will be located as follows: (1) 10 soil borings will be located in the north area of the OU2 M&H Site; (2) 10 soil borings will be located in the northeast periphery of the OU2 M&H Site (east of the Central Railroad and west of the Little Vermilion River); (3) 10 soil borings will be located in the main industrial area; (4) 10 soil borings will be located around Building 100; (5) 10 soil borings will be located in the exterior northwest corner of the rolling mill; and (6) 10 soil borings will be installed inside the rolling mill (see Figure 2).

Fifty of these soil borings will be advanced to a depth of 12 feet below ground surface (bgs) unless refusal is encountered before 12 feet. The remaining 10 soil borings (near Building 100) will be advanced to a depth greater than 12 feet bgs depending on PCB concentrations in an area of known PCB contamination at 12 feet bgs. In this known PCB contamination area, near Building 100, SulTRAC will be using PCB-specific chemistry field kits to determine PCB concentrations at depths greater than 12 feet. The goal is to continue Geoprobe activities with depth until no PCBs are detected via field kit results. This "clean" deep horizon will then be sampled and sent to the CLP laboratory for verification.

All the surface and subsurface samples from the combined 30 soil borings in the north area, northeast periphery area, and inside the rolling mill will be analyzed for TAL metals (including mercury) and cyanide, VOCs, SVOCs, PCBs, and pesticides; only these surface samples will also be analyzed for asbestos. All the surface and subsurface samples from the 10 soil borings located in the main industrial area will be sampled for metals and undergo the synthetic precipitation leaching procedure (SPLP) with five samples also being analyzed via the toxicity characteristic leaching procedure (TCLP). Samples from the 10 soil boring locations in the exterior northwest corner of the rolling mill will be analyzed for VOCs, and TAL metals (including mercury) and cyanide. The 10 soil borings located by Building 100 will be analyzed for PCBs, and TAL metals (including mercury) and cyanide. Soil boring samples will be continuously collected using direct-push technology. Of the above listed soil boring

locations, 10 will be advanced by an all-terrain vehicle-mounted Geoprobe®, and an additional 10 borings will be drilled by hand-augering or using a hand Geoprobe®. Samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, metals, TCLP metals, and SPLP metals.

During the Phase II field investigation, SulTRAC will coordinate with the EPA's FIELDS team in screening approximately 200 surface soil locations with an Innov-X XRF analyzer in an unbiased sampling approach using site sampling grids combined with EPA's rapid assessment tools (RAT). The purpose of the XRF field program is to gain extensive and high spatial resolution metals concentrations for the metals of interest (based on the Phase I results)—arsenic, cadmium, lead, mercury, and zinc. This is particularly important in areas difficult for Geoprobe to access, and for sampling in an unbiased manner during Phase II in order to determine contamination extent. SulTRAC will collect approximately 50 samples from these screened XRF locations for analytical measurement via CLP laboratory. These 50 samples will be analyzed for TAL metals (including mercury) and cyanide. These fixed lab results will be calibrated against the XRF results at the 50 locations from which the samples had been collected for fixed-lab analysis, creating a calibration curve against which all OU2 M&H Site XRF data can be matched.

SulTRAC will collect 50 building material samples from building structures throughout the OU2 M&H Site to be analyzed for VOCs, SVOCs, PCBs, pesticides, TAL metals (including mercury) and cyanide, and TCLP metals (see Figure 3).

Seventeen new monitoring wells and six piezometers will be installed during July and August 2008 (see Figure 4). The 17 new monitoring wells will be developed and advanced to depths ranging approximately from 13 to 44 feet bgs. Six monitoring wells will be installed around the rolling mill to further delineate TCE contamination; one monitoring well will be installed near Building 100 to further delineate PCB contamination; 7 monitoring wells will be installed in the former main processing area; 2 monitoring wells will be installed in the northern area of the OU2 M&H Site, and 1 additional monitoring well will be installed on the western perimeter of the site. The groundwater sampling program will include a total of 7 quarters of groundwater sampling, of which 6 of the seven events will include both Phase I (19) and Phase II (17) monitoring wells. Samples will be collected from all 36 wells for analysis for VOCs, SVOCs, pesticides, PCBs, and TAL metals (including mercury) and cyanide during the four initial quarters. After the first four initial quarterly sampling events, analytical data will be evaluated to determine the chemicals of interest for each well. A technical memorandum will be submitted to the EPA detailing the limited analyte groups (chemicals of interest) that will be sampled for the second year of groundwater sampling (four quarters). SulTRAC assumes that for Phase I and Phase II second round of quarterly of sampling all monitoring wells will be sampled for TAL metals (including mercury) and cyanide, and one-third of the monitoring wells will be sampled for the other analyte groups (VOC, SVOC, pesticides, PCBs). The criteria used to select the monitoring well locations included geographic spread, expected high and low contaminant concentrations, location, and groundwater depth. Groundwater samples will be collected from the installed and developed monitoring wells constructed of PVC casing and a PVC 10-foot, 10 slot screen.

During the Phase II field investigation, SulTRAC will also collect surface water samples on two separate days. Two hydrologic investigations will be conducted to sample surface water at the beginning of summer and at the end of the summer 2008. We are testing seasonal variations, as precipitation variation exerted no influence during Phase I. Eight samples will be collected during each sampling event (see Figure 5). Three drainage pipes discharging water were observed in the northeast corner of the site during a site visit in April 2008. Surface water samples will be collected at these three locations, at two locations at the mouth and terminus of the creek emanating from the abandoned sewer line and emptying into the Little Vermilion River, at one location in the north area where standing water is often witnessed, and at two locations of discharge from the

main industrial plant area. Surface water samples will be analyzed for VOCs, SVOCs, PCBs, pesticides, and filtered and unfiltered TAL metals (including mercury) and cyanide, and total hardness.

SulTRAC will conduct ecological (bioavailability sampling) and biological (bioassessibility sampling) investigations. Bioavailability sampling will include aboveground and belowground vegetation sample pair samples as well as soil invertebrate sampling from areas of ecological interest in OU2. If enough biomass cannot be collected, a contingency plan has been developed for SulTRAC to collect surface soil, sinter, and slag samples to submit to a laboratory bioavailability testing for vegetation (lettuce) and earthworms. Bioassessibility sampling will include soil, sinter, and slag sample collection throughout OU2. SulTRAC will analyze data from the soil, solids, surface water, and groundwater samples for the analytical groups listed above to delineate the contamination present at the OU2 M&H Site.

QAPP WORKSHEET #18
SAMPLING LOCATIONS/IDS, SAMPLE DEPTHS, SAMPLE ANALYSES AND SAMPLING PROCEDURES TABLE

(UFP QAPP Section 3.1.1)
List all locations that will be sampled indicating the sample identity (ID) number or sample location. Specify sample matrix and depth at which samples will be taken. List all analyses the samples will be analyzed for.
Specify the appropriate SOP or specific section in the SAP that describes the sample collection procedure.

Sampling Location[1]/ ID Number	Matrix	Depth (feet bgs)	Analytical Group	Sampling SOP Reference[2]
4 locations	Air	NA	NIOSH Method 7402 (asbestos)	S-1, S-2
30 locations (two depths each)	Soil[3]	0-2 and 2-12 or refusal	CLP SOW SOM01.2 (VOA, SVOA, PCBs, and pesticides) CLP SOW ILM05.4 (TAL metals, mercury, cyanide) EPA Method 600/R-93-116 (asbestos) (0 to 2-foot bgs depth only)	S-3, S-4, S-5, S-17
10 locations (two depths each)	Soil[3]	0-2 and 2-12 or refusal	CLP SOW ILM05.4 (TAL metals, mercury, cyanide) EPA SW-846 Method 1312 and CLP SOW ILM05.4 (SPLP metals) EPA SW-846 Method 1311 and CLP SOW ILM05.4 (TCLP metals) (10 depths only)	S-3, S-4, S-5
10 locations (two depths each)	Soil[3]	0-2 and >12	CLP SOW SOM01.2 (PCBs) CLP SOW ILM05.4 (TAL metals, mercury, cyanide)	S-3, S-4, S-5, S-18
10 locations (two depths each)	Soil[3]	0-2 and 2-12 or refusal	CLP SOW SOM01.2 (VOA) CLP SOW ILM05.4 (TAL metals, mercury, cyanide)	S-3, S-4, S-5
50 locations	Soil[3]	Surface	CLP SOW ILM05.4 (TAL metals, mercury, cyanide)	S-3, S-7
12 locations	Vegetation	Surface	SOW ILM05.4 (TAL metals, mercury)	S-20
20 locations	Soil invertebrates	Surface	ASTM Method E 1676-04 (bioavailability)	S-3
40 locations (contingency)	Soil	Surface	ASTM Method E 1676-04 (bioavailability)	S-3
11 locations	Soil (sinter, slag, or soil)	Surface	Relative bioassessibility leaching procedure	S-16
50 locations	Solids/building materials	Surface	CLP SOW SOM01.2 (VOA, SVOA, PCBs, and pesticides) CLP SOW ILM05.4 (TAL metals, mercury, cyanide) EPA SW-846 Method 1311 (TCLP metals) EPA Method 600/R-93-116 (asbestos)	S-6, S-17

QAPP WORKSHEET #18 (CONTINUED)
SAMPLING LOCATIONS/IDS, SAMPLE DEPTHS, SAMPLE ANALYSES AND SAMPLING PROCEDURES TABLE

Sampling Location[1]/ ID Number	Matrix	Depth (feet bgs)	Analytical Group	Sampling SOP Reference[2]
8 locations (two sampling events)	Surface water	Surface	CLP SOW SOM01.2 (VOA, SVOA, PCBs, and pesticides) CLP SOW ILM05.4 (TAL metals, mercury, cyanide) EPA Method 130.1 (hardness)	S-15, S-20, S-21
36 locations	Groundwater[4]	13 to 46	CLP SOW SOM01.2 (VOA, SVOA, PCBs, and pesticides) CLP SOW ILM05.4 (TAL metals, mercury, cyanide)	S-12, S-13

Notes:

ASTM American Society of Testing Materials
ID Identification
NA Not applicable
NIOSH National Institute for Occupational Safety and Health
SW Solid waste

1 See Figures 2 to 5 for sampling locations and Table 2 for specific sample identification numbers.
2 See Worksheet #21 for a list of sampling methods S-1 through S-19
3 Samples will be collected from soil borings.
4 Samples will be collected from 36 monitoring wells for the six quarters of groundwater sampling events scheduled for the Phase II field investigation.

QAPP WORKSHEET #19
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

(UFP QAPP Section 3.1.1)
For each matrix and analytical group list the analytical and preparation method and associated container specifications, preservation requirements, and maximum holding time.

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis)[1]
Air	Asbestos	NIOSH 7402	25-mm-diameter, three-piece cassette loaded with an MCE filter of pore size 0.45 µm; filter should be backed by a 5-µm pore size MCE filter	Not required	Not required
Soil		EPA 600/R-93-116	One 8-ounce wide-mouth glass jar	Not required	180 days (from the date of sampling)
Solids		EPA 600/R-93-116	Oversized concrete, wood, or stone samples – Place entire sample in ziplock-type plastic bag. Instruct laboratory to collect a sub-sample. Rubble: One 4-, 8-, or 16-ounce wide-mouth glass jar Container volume depends on matrix size	Not required	180 days (from the date of sampling)
Soil	VOCs	CLP SOW SOM01.2	Three 40-mL glass containers with PTFE-lined septa and open-top screw caps, pre-weighed and containing magnetic stir bars and one container of sample filled with no headspace for determination of moisture content At least three coring tools used as transport devices (for example, 5-gram samplers) and one container of sample filled with no headspace for determination of moisture content	Cool to 4 °C ± 2 °C immediately after collection Frozen (-7 °C to -15 °C)	48 hours to preservation at laboratory/14 days for analysis following preservation 48 hours (frozen) to preservation at laboratory for analysis after preservation

QAPP WORKSHEET #19 (CONTINUED)
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis)[1]
Solids	VOCs	CLP SOW SOM01.2	Granular Solids – Three 40-mL glass containers with PTFE-lined septa and open-top screw caps, pre-weighted and containing magnetic stir bars and one container of sample filled with no headspace for determination of moisture content Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	48 hours to preservation at laboratory/14 days for analysis following preservation
Soil	SVOCs	CLP SOW SOM01.2	Two 4-ounce or one 8-ounce wide-mouth glass jar	Cool to 4 °C ± 2 °C immediately after collection	14 days/40 days
Solids		CLP SOW SOM01.2	Granular Solids – Two 4-ounce or one 8-ounce wide-mouth glass jars depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	14 days/40 days
Soil	PCBs	CLP SOW SOM01.2	Two 4-ounce or one 8-ounce wide-mouth glass jar	Cool to 4 °C ± 2 °C immediately after collection	14 days/30 days
Solids		CLP SOW SOM01.2	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	14 days/30 days

QAPP WORKSHEET #19 (CONTINUED)
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis)[1]
Soil	Pesticides	CLP SOW SOM01.2	Two 4-ounce or one 8-ounce wide-mouth glass jar	Cool to 4 °C ± 2 °C immediately after collection	14 days/40 days
Solids		CLP SOW SOM01.2	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Wrap entire sample in aluminum foil and place in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	14 days/40 days
Soil	Metals (including mercury and cyanide)	CLP SOW ILM05.4	Two 4-ounce or one 8-ounce wide-mouth glass jar	Cool to 4 °C ± 2 °C immediately after collection	NA/6 months (Metals and Mercury) 14/14 days (Cyanide)
Solids		CLP SOW ILM05.4	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Place sample in ziplock-type plastic bag. Instruct CLP lab to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	NA/6 months
Soil	TCLP Metals	EPA SW-846 Method 1311[2]	Two 4-ounce or one 8-ounce wide-mouth glass jar.	Cool to 4°C ± 2°C immediately after collection	180 days to TCLP extraction/180 days to analysis
Solids		EPA SW-846 Method 1311[2]	Granular Solids – Two 4- or 8-ounce wide-mouth glass jars, depending on size of matrix. Granularize and homogenize as much as possible. Oversized concrete, wood, or stone samples – Place sample in ziplock-type plastic bag. Instruct CLP laboratory to collect a sub-sample.	Cool to 4 °C ± 2 °C immediately after collection	180 days to TCLP extraction /180 days to analysis

QAPP WORKSHEET #19 (CONTINUED)
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis) [1]
Soil	SPLP Metals	EPA SW-846 Method 1312[2]	Two 4-ounce or one 8-ounce wide-mouth glass jar	Cool to 4 °C ± 2 °C immediately after collection	180 days from collection to extraction/180 from extraction to analysis 28 days for mercury
Vegetation (*in-situ* bioavailability study)	Metals	SOW ILM05.4 Sava 1994	1-gallon Ziploc bags	Send to laboratory on dry ice	24 hours to dry ice/NC
Soil Invertebrates (*in situ* bioavailability study)	Metals	ASTM Method E 1676-04	1-gallon Ziploc bags	Rinse with de-ionized water and purge earthworms for 24 hours Send samples to laboratory on dry ice.	24 hours for purge and dry ice/NC
Soil (contingency-bioavailability study)	Metals	ASTM Method E 1676-04	3-gallon container	Cool to 4 °C ± 2 °C immediately after collection	14 days survival 28 days bioavailability
Soil (vegetation study)	Metals	ASTM Method E 1598-94	1-gallon Ziploc bags	Cool to 4 °C ± 2 °C immediately after collection	28 days (refrigerated) or 6 month (if frozen)
Soil (bioassess-ibility study)	Lead and arsenic	Relative Bioavailability Leaching Procedure	8-ounce wide-mouth glass jars	Cool to 4 °C ± 2 °C immediately after collection	NA/6 months
Water	VOCs	CLP SOW SOM01.2	Three 40-mL glass vials with PTFE-lined septa and open-top screw caps	No headspace Cool to 4 °C ± 2 °C Adjust pH to less than 2 with HCl	7 days/14 days
Water	SVOCs	CLP SOW SOM01.2	Two 1-liter amber glass bottles fitted with PTFE-lined screw caps	Cool to 4 °C ± 2 °C immediately after collection; keep away from light	7 days/40 days

QAPP WORKSHEET #19 (CONTINUED)
ANALYTICAL METHODS, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Containers (number, size, type)	Preservation Requirements (chemical, temperature, etc.)	Maximum Holding Time (preparation/analysis) [1]
Water	PCBs	CLP SOW SOM01.2	Two 1-liter amber glass bottles fitted with PTFE-lined screw caps	Cool to 4 °C ± 2 °C immediately after collection; keep away from light	7 days/40 days
Water	Pesticides	CLP SOW SOM01.2	Two 1-liter amber glass bottles fitted with PTFE-lined screw caps	Cool to 4 °C ± 2 °C immediately after collection; keep away from light	7days/40 days
Water	TAL Metals, Mercury	CLP SOW ILM05.4	One 1-liter high-density polyethylene bottle One 1-liter high-density polyethylene bottle with 0.45µm filter for filtered surface water sample	HNO_3 to pH < 2 and cool to 4 °C (±2 °C) immediately after collection	NA/6 months (Metals) NA/28 days (Mercury)
Water	TAL Cyanide	CLP SOW ILM05.4	One 1-liter high-density polyethylene bottle	NaOH to pH>12 and cool to 4 °C (±2 °C) immediately after collection	NA/14 days
Water	Total Hardness	EPA Method 130.1	One 500-mL high-density polyethylene bottle	HNO_3 to pH < 2 and cool to 4 °C (±2 °C) immediately after collection	NA/6 months

Notes:

µm Micrometer
HCl Hydrochloric acid
HNO_3 Nitric acid
MCE Mixed cellulose ester
mL Milliliter
mm Milliliter
NA Not applicable
NaOH Sodium hydroxide
PTFE Polytetrafluoroethylene

1 Holding time is applicable from validated time of sample receipt and is measured to time of sample extraction and analysis.
2 TCLP and SPLP extraction procedures listed; analytical method for leachate TBD

QAPP WORKSHEET #20
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

(UFP QAPP Section 3.1.1)
Summarize by matrix and analytical group.

Matrix	Analytical Group	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Samples	No. of Field Duplicates[2]	No. of MS/MSDs[3]	No. of Trip Blanks[4]	No. of Equipment Rinsates	Total No. of Samples to Laboratory
Soil	VOA/CLP	A-1	40	80	8	4	5	2	99
Soil	SVOA/CLP	A-1	30	60	6	3	0	2	71
Soil	PCBs/CLP	A-1	40	80	8	4	0	2	94
Soil	Pesticides/CLP	A-1	30	60	6	3	0	2	71
Soil	TAL Metals, Mercury, Cyanide/CLP	A-2	60	120	12	6	0	2	140
Soil	TCLP Metals / CLP	A-3	5	10	1	1	0	0	12
Soil	SPLP Metals / CLP	A-4	10	20	2	1	0	0	23
Soil (from XRF screening event)	Metals	A-2	200	50	5	3	0	0	58
Vegetation (bioavailability)	Metals	A-2	12	12	0	0	0	0	12
Soil Invertebrates (bioavailability)	Metals	A-5	4	20	0	0	0	0	20
Soil (bioavailability) (contingency)	Metals	A-5	40	40	4	0	0	0	44
Soil (bioassessibility)	Lead and Arsenic	A-7	11	11	1	0	0	0	12
Soil[5]	Asbestos	A-8	30	40	3	0[6]	0	0	43
Air	Asbestos	A-9	4	4	0	0	0	0	4
Solid/Building Material	Asbestos	A-8	50	50	5	0[6]	0	0	55
Solid/Building Material	VOA/CLP	A-1	5	5	0	0	2	0	12

QAPP WORKSHEET #20 (CONTINUED)
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

Matrix	Analytical Group	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Samples	No. of Field Duplicates[2]	No. of MS/MSDs[3]	No. of Trip Blanks[4]	No. of Equipment Rinsates	Total No. of Samples to Laboratory
Solid/Building Material	SVOA/CLP	A-1	50	50	5	3	0	0	58
Solid/Building Material	PCB/CLP	A-1	50	50	5	3	0	0	58
Solid/Building Material	Pesticide/CLP	A-1	50	50	5	3	0	0	58
Solid/Building Material	TAL Metals, Mercury, Cyanide/ CLP	A-2	50	50	5	3	0	0	58
Solid/Building Material	TCLP Metals /CLP	A-3	5	5	0	0	0	0	5
Surface Water[7]	VOA/CLP	A-1	8	16	2	2	2	0	22
Surface Water[7]	SVOA/CLP	A-1	8	16	2	2	0	0	20
Surface Water[7]	PCB/CLP	A-1	8	16	2	2	0	0	20
Surface Water[7]	Pesticide/CLP	A-1	8	16	2	2	0	0	20
Surface Water[7]	TAL Metals, Mercury/ CLP	A-2	8	16	2	2	0	0	20
Surface Water[7]	TAL Cyanide /CLP	A-2	8	16	2	2	0	0	20
Surface Water[7]	Total Hardness	A-10	8	8	1	0	0	0	9
Groundwater[8]	VOA/CLP	A-1	36	154	16	18	10	18	216
Groundwater[8]	SVOA/CLP	A-1	36	154	16	18	0	18	206
Groundwater[8]	PCB/CLP	A1	36	154	16	18	0	18	206
Groundwater[8]	Pesticide/CLP	A-1	36	154	16	18	0	18	206
Groundwater[8]	TAL Metals, Mercury/CLP	A-2	36	250	25	18	0	18	206
Groundwater[8]	TAL Cyanide /CLP	A-2	36	154	16	18	0	18	206

QAPP WORKSHEET #20 (CONTINUED)
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

Notes:

Sample numbers in this table reflect field QC samples collected during each sampling event.

1 Analytical and preparation SOPs are listed in Worksheet #23.
2 Field duplicates are collected at a rate of 1 per 10 investigative samples of the same matrix.
3 MS/MSD samples are collected at a rate of 1 per 20 investigative samples of the same matrix.
4 A trip blank will be provided with each shipping container to be analyzed for VOCs.
5 Solid asbestos samples include 30 soil samples and 10 performance evaluation samples.
6 No MS/MSD samples will be collected for asbestos soils/solids.
7 Surface water samples will be collected at the beginning and the end of Summer 2008.
8 The table presents the minimum number of primary sampling locations. The installed Phase I monitoring wells (MW1 through MW18) will be sampled for a reduced analyte list beginning the Winter 2008 groundwater sampling event. The Phase I reduced list will be decided after the Fall 2008 groundwater sampling event after a technical memorandum (#1) (Phase I wells only) is submitted to the EPA detailing the initial four quarters of sampling results from MW1 through MW18. Phase II monitoring wells (MW19 through MW35) will be sampled for a reduced analyte list beginning in the Summer 2009 event. The Phase II reduced list will be decided after the Spring 2009 groundwater sampling event after a technical memorandum (#2) (Phase II only) is submitted to the EPA detailing the initial four quarters of sampling results from MW19 through MW35.

QAPP WORKSHEET #21
PROJECT SAMPLING SOP REFERENCES TABLE

(UFP Section 3.1.2)
List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody. Include copies of the SOPs as attachments or reference all in the QAPP. Sequentially number sampling SOP references in the Reference Number column. The reference number can be used throughout the QAPP to refer to a specific SOP.

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
S-1	Air Quality Monitoring, Revision No. 0, November 1999, SOP 073	Tetra Tech EM Inc.	Air sampler, asbestos filter, CMS cartridge, or a GC adsorbent cartridge	N	None
S-2	Calibration of Air Sampling Pump, Revision No. 0, November 1999	Tetra Tech EM Inc.	Air sampling pump, digital calibrator (soap bubble meter), soap solution temperature and pressure gauge	N	None
S-3	Soil Sampling, Revision No. 1, December 1999, SOP 005	Tetra Tech EM Inc.	Spoon or spatulas, trowel, split-spoon sampler, coring tools	N	None
S-4	Using the Geoprobe System, Revision No. 1, December 1999, SOP 054	Tetra Tech EM Inc.	Shelby tube drive head, probe drive Geoprobe Systems	N	None
S-5	Sludge and Sediment Sampling, Revision No. 3, January 2000, SOP 006	Tetra Tech EM Inc.	Sample containers, stainless-steel scoop or trowel, hand corer, Ponar grab sampler	N	None

QAPP WORKSHEET #21 (CONTINUED)
PROJECT SAMPLING SOP REFERENCES TABLE

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
S-6	Bulk Material Sampling, Revision No. 3, December 1999, SOP 007	Tetra Tech EM Inc.	Sample containers, stainless-steel scoop, trowel, coring tools	N	None
S-7	Field Portable Innov-X XRF Spectrometry for the Determination of Elemental Concentrations in Soil, Revision 3, February, 2007, XRF SOP	EPA	Field Portable Innov-X XRF Analyzer	N	None
S-8[1]	Monitoring Well Installation, Revision No. 3, December 2000, SOP 020	Tetra Tech EM Inc.	Casing materials, well screen materials, filter pack materials, annular sealant, grouting materials, tremie pipe, surface completion and protective casing materials, concrete surface pad and bumper post, uncontaminated water	N	None
S-9	Borehill Drilling-Hollow Stem Auger Drilling, Revision No. 1, March 1992, SOP 045	Tetra Tech EM Inc.	Hollow stem auger drill rig (with associated drill tools and hardware)	N	None
S-10[1]	Monitoring Well Development, Revision No. 3, October 2000, SOP 021	Tetra Tech EM Inc.	Pumps, air compressors, bailers, surge blocks	N	None
S-11	Static Water Level, Total Well Depth, and Immiscible Layer Measurement, Revision No. 0, December 1999, SOP 014	Tetra Tech EM Inc.	Electrical water level indicator, interface probe, PID or FID	N	None

QAPP WORKSHEET #21 (CONTINUED)
PROJECT SAMPLING SOP REFERENCES TABLE

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
S-12	Groundwater Sample Using Micropurge Technology, Revision No. 1, January 2000, SOP 015	Tetra Tech EM Inc.	Water level indicator, adjustable flow rate pump, discharge flow controller, flow-through cell, pH probe, dissolved oxygen probe, turbidity meter, oxidation and reduction probe, containers	N	None
S-13	Groundwater Sampling Revision No. 3 March 2000 SOP 010	Tetra Tech EM Inc.	Sample bottles, high-density polyethylene bailer, or peristaltic pumps	N	None
S-14	Slug Test, Draft, June 1995, SOP Slug Test	Sullivan International Group	Slug, an electronic data logger, pressure transducer, conductor cable	N	None
S-15	Surface Water Sampling, Revision No. 3, December 1999, SOP 009	Tetra Tech EM Inc.	Sample bottles, dipper, or other device made of inert material (stainless steel or Teflon)	N	None
S-16	The In-Vitro Method, Relative Bioavailability Leaching Procedure, 2003	Laboratory for Environmental and Geological Studies (LEGS)	Extraction device, wide-mouth HDPE bottles, Plexiglas tank, circulator heater	N	None
S-17	Asbestos Sampling, Revision No. 0, April 2003, SOP S014	Sullivan International Group	Sample containers with Teflon-lined lids or Ziploc bags, coring tools	N	None
S-18	Test Method for Polychlorinated Biphenyls in Soil, Revision No. 0, December 1996	EPA	L2000 PCB/chloride analyzer or equivalent	N	None
S-19[1]	General Equipment Decontamination, Revision No. 2, December 1999, SOP 002	Tetra Tech EM Inc.	Scrub brushes, large wash tubs or buckets, Alconox, distilled water	N	None

QAPP WORKSHEET #21 (CONTINUED)
PROJECT SAMPLING SOP REFERENCES TABLE

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
S-20	Sava, Roger. 1994. "Guide to Sampling Air, Water, Soil, and Vegetation for Chemical Analysis." California Environmental Protection Agency. Environmental Hazards Assessment Program, State of California. June.	California Environmental Protection Agency. Environmental Hazards Assessment Program	Stainless steel sheers and stainless steel trowels, ziplock bags	N	None
S-21	US EPA Region 6. Water-Quality Samples for Dissolved Metals-in-Water, SOP No. , Revision 0. January 13, 2000.	US EPA Region 6	Tygon tubing, pump, 0.45μm filter, 1-L HDPE bottle	N	None

Notes:

CMS Carbon molecular sieve
HDPE High-density polyethylene
PID Photoionization detector
FID Flame ionization detector
GC Gas chromatograph

1 SulTRAC will use these non-sampling SOPs for field activities other than sampling as specified in the SOP.

QAPP WORKSHEET #22
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP QAPP Section 3.1.2.4)

Identify all field equipment/instruments that require calibration, maintenance, testing, or inspection activities. Specify the frequency of each activity, acceptance criteria, and corrective action requirements. Provide the SOP reference number for each type of equipment, if available.

Field Equipment	Calibration Activity[1]	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference	Comments
Multiparameter Water Quality Meter[2]	Oxidation-reduction potential: 2 standard solutions pH: 2 standard solutions Conductivity: 1 standard solution Temperature: no standard solution Turbidity: 2 standard solutions Dissolved oxygen: 2 standard solutions	Daily before first field measurement and after final field measurement	± 10 millivolts ± 0.01 pH unit ± 3% ± 0.1 °C ± 10% ± 10%	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	F-1 (Field Measurement of Water Temperature, SOP 011, Revision No. 2, November 1999) F-2 (Field Measurement of pH, SOP 012, Revision No. 3, November 1999) F-3 (Field Measurement of Specific Conductance, SOP 013, Revision No. 2, November 1999)	See note below
PID	Gas calibration standard or equivalent	Daily before first field measurement	10% of reading < 2,000 ppm 20% of reading > 2,000 ppm	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	F-4 (Field Measurement of Organic Vapor Air, SOP 003, Revision No. 2, December 1999)	None

QAPP WORKSHEET #22 (CONTINUED)
FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

Field Equipment	Calibration Activity[1]	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference	Comments
High-volume air sampling pump	Per manufacturer's instructions	Daily before first field measurement and after final field measurement	Flow rate: 0.5 to 16 L/min; minimum recommended volume is 400 L at 0.1 fiber/cc	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	F-5 (Air Quality Monitoring, SOP 073, Revision No. 1, November 1999)	None
Innov-X XRF Analyzer	Per manufacturer's instructions	Daily before first field measurement	Standard results must be within ± 30% of true value	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	F-6 (X-ray Fluorescence Spectrometry for the determination of Elemental Concentrations in Soil, Revision No.3, February 2007)	None
L2000DX Analyzer System (or equivalent)	Per manufacturer's instructions	Daily before first field measurement	Soil: 2- 2,000 ppm	Repeat calibration; correct measurements for drift if necessary	Field team leader or field team members	F-7 (Test Method for Polychlorinated Biphenyls in Soil, Revision No.0, December 1996)	None

Notes:

SulTRAC will measure water temperature, pH, and specific conductance in purged groundwater until these parameters have stabilized.

cc Cubic centimeter
L/min Liter per minute
ppm Part per million

1 The field equipment will be calibrated per manufacturer's instructions.
2 Standard solutions will be provided by the vendor to calibrate this instrument.

QAPP WORKSHEET #23
ANALYTICAL SOP REFERENCES TABLE

(UFP QAPP Section 3.2.1)
List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP references in the reference number column. The reference number can be used throughout the QAPP to refer to a specific SOP. Include copies of the SOPs as attachments or reference in the QAPP.

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
A-1	CLP SOW SOM01.2 for Organics Analysis, Multi-Media, Multi-Concentration	Definitive	VOA, SVOA	GC/mass spectroscopy	CLP Laboratory	No
A-1	CLP SOW SOM01.2 for Organics Analysis, Multi-Media, Multi-Concentration	Definitive	PCB, pesticide	GC/electron capture detector	CLP Laboratory	No
A-2	CLP SOW ILM05.4 for Inorganic Analysis, Multi-Media, Multi-Concentration	Definitive	TAL Metals, Mercury, and Cyanide	ICP/AES ICP/mass spectroscopy Cold vapor atomic absorption	CLP Laboratory	No
A-3[1]	EPA SW-846, Method 1311, Method for Toxicity Characteristic Leaching Procedure	Definitive	TCLP Metals	ICP/AES ICP/mass spectroscopy Cold vapor atomic absorption	CLP Laboratory	No
A-4[1]	EPA SW-846, Method 1312, Method for Synthetic Precipitation Leaching Procedure	Definitive	SPLP Metals	ICP/AES	CLP Laboratory	No
A-5	ASTM Method E 1676-04, Method for Conducting Laboratory Soil Toxicity or Bioaccumulation for Soil Invertebrates	Definitive	NA	NA	Test America	No

QAPP WORKSHEET #23 (CONTINUED)
ANALYTICAL SOP REFERENCES TABLE

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
A-6	ASTM Method E 1598-94, Method for Conducting Early Seedling Growth Tests	Definitive	NA	NA	Subcontracted Laboratory	No
A-7	Relative Bioavailability Leaching Procedure	Definitive	Lead and Arsenic	TBD	LEGS	No
A-8	EPA Method 600/R-93-116, Method for the Determination of Asbestos in Bulk Building Materials	Definitive	Asbestos	Polarized light microscopy	STAT Analysis Corporation	No
A-9	NIOSH Method 7402, Method for the Determination of Asbestos in Ambient Air	Definitive	Asbestos	Transmission electron microscopy	STAT Analysis Corporation	No
A-10	EPA Method 130.1, Hardness, total (mg/L as $CaCO_3$) (Colorimetric, Automated EDTA)	Definitive	Hardness (Surface Water)	Spectrophotometer	CLP Modified Analysis	No

Notes:

AES Atomic emission spectroscopy
$CaCO_3$ Calcium carbonate
EDTA
ICP Inductively coupled plasma
NA Not applicable

1 TCLP and SPLP extraction procedures listed; analytical method for leachate is A-2, CLP SOW ILM05.4 for Inorganic Analysis, Multi-Media, Multi-Concentration.

QAPP WORKSHEET #24
ANALYTICAL INSTRUMENT CALIBRATION TABLE

(UFP Section 3.2.2)
Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference[1]
GC/Mass Spectroscopy	VOCs: Run five calibration standard solutions and a blank SVOCs: Run five calibration standard solutions and a blank	12-hour continuing calibration acceptance criteria	Always, RRF $\geq$ 0.010 or per SOP Initial, RSD $\leq$ 20% or 40%, depending on compound. Continuing, %D $\leq$ 25 or 40 depending on compound	Inspect the system for problems, clean the ion source, change the column, service the purge and trap device, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-1
GC/Electron Capture Detector	Pesticides: Run five calibration standard solutions and a blank PCBs: Run five calibration standard solutions and a blank	12-hour continuing calibration acceptance criteria	Always, resolution per SOP Initial, CF RSD $\leq$ 20% Continuing, CF %D $\leq$ 15 for opening and $\leq$ 50 for closing	Inspect the system for problems, change the column, bake out the detector, clean the injection port, and take other CAs to achieve the acceptance criteria.	CLP Laboratory Analyst	A-1
ICP/AES	Run five calibration mixed standard solutions and a blank	Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV)	Deviation from the initial calibration verification: metals 90-110%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-2

QAPP WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION TABLE

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference[1]
ICP/Mass Spectroscopy	Run at least six calibration standard solutions and three blanks	Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV)	Deviation from the initial calibration verification: metals 90-110%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-2
ICP/AES	Run five calibration mixed standard solutions and a blank	Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV)	Deviation from the initial calibration verification: metals 90-110%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-3
ICP/AES	Run five calibration mixed standard solutions and a blank	Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV)	Deviation from the initial calibration verification: metals 90-110%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-4
Polarized Light Microscopy	NA	A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained	<1% to 100%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Subcontracted Laboratory Analyst	A-8

QAPP WORKSHEET #24 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION TABLE

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference[1]
Transmission Electron Microscopy	NA	NA	0.04 to 0.5 fiber/cc for a 1,000-liter air sample	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Subcontracted Laboratory Analyst	A-9
Spectrophotometer Technicon AutoAnalyzer	Run five calibration mixed standard solutions and a blank	Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV)	Deviation from the initial calibration verification: metals 90-110%	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-10

Notes:

%D Percent difference
CCV Continuing calibration verification
CF Calibration factor
NA Not available
RRF Relative response factor
RSD Relative standard deviation

1 See Worksheet #23 for analytical methods.

QAPP WORKSHEET #25
ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE TESTING, AND INSPECTION TABLE

(UFP QAPP Section 3.2.2)
Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument/ Equipment	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
GC/Mass Spectroscopy	Daily Check, Instrument tune (4-bromofluorobenzene or decafluorotriphenylphosphine)	Injector syringe, injector septum, injector liner/seal, injector port, guard column, column splitter, analytical column, ion source, detector, traps, and gas supply	See A-1	See A-1	Inspect the system for problems, clean the ion source, change the column, and service the purge and trap device.	CLP Laboratory Analyst	A-1
GC/Electron Capture Detector	Daily Check, Initial Calibration Verification	Injector syringe, injector septum, injector liner/seal, injector port, guard column, column splitter, analytical column, ion source, detector, traps, and gas supply	See A-1	See A-1	Inspect the system for problems, change the column, bake out the detector, and clean the injection port.	CLP Laboratory Analyst	A-1
ICP/AES	Daily Check, Initial Calibration Verification	Nebulizer, injection tube, flame optimization, gas supply, and detector	See A-2	See A-2	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria	CLP Laboratory Analyst	A-2

QAPP WORKSHEET #25 (CONTINUED)
ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE TESTING, AND INSPECTION TABLE

Instrument/ Equipment	Maintenance Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
ICP/Mass Spectroscopy	Daily Check, Initial Calibration Verification	Nebulizer, injection tube, plasma optimization, gas supply, and detector	See A-2	See A-2	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP Laboratory Analyst	A-2
Polarized Light Microscopy	Daily Check	Frequency of inspection depends on equipment	See A-8	See A-8	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Subcontracted Laboratory Analyst	A-8
Transmission Electron Microscopy	Daily Check	Frequency of inspection depends on equipment	See A-9	See A-9	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	Subcontracted Laboratory Analyst	A-9
Spectrophotometer Technicon AutoAnalyzer	Daily Check	Frequency of inspection depends on equipment	See A-10	See A-10	Inspect the system for problems, clean the system, verify operating conditions, and take CAs to achieve the technical acceptance criteria.	CLP or CRL Laboratory Analyst	A-10

Note:

1 See Worksheet #23 for identification of analytical methods.

QAPP WORKSHEET #26
SAMPLE HANDLING SYSTEM

(UFP QAPP Appendix A)
Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field sampling personnel/SulTRAC
Sample Packaging (Personnel/Organization): Field sampling personnel/SulTRAC
Coordination of Shipment (Personnel/Organization): Field sampling personnel/SulTRAC
Type of Shipment/Carrier: Cooler packed with ice and packing material such as bubble wrap/FedEx or other overnight courier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory personnel/CLP laboratory and subcontracted laboratory
Sample Custody and Storage (Personnel/Organization): Laboratory personnel/CLP laboratory and subcontracted laboratory
Sample Preparation (Personnel/Organization): Laboratory personnel/CLP laboratory and subcontracted laboratory
Sample Determinative Analysis (Personnel/Organization): Laboratory personnel/CLP laboratory and subcontracted laboratory
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): See Worksheet #27
SAMPLE DISPOSAL
Personnel/Organization: Laboratory personnel/CLP laboratory and subcontracted laboratory
Number of Days from Analysis: TBD (or in accordance with individual laboratory SOP)

QAPP WORKSHEET #27
SAMPLE CUSTODY REQUIREMENTS

(UFP Appendix A)
Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain of custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory): SulTRAC will use EPA's Field Operations and Records Management System (FORMS II Lite) software to manage sample collection, documentation, chain of custody, and reporting. Field personnel will input data into FORMS II Lite and then use the software to generate sample labels, bottle tags, and chain-of-custody forms to track samples from the field to the laboratory. Because FORMS II Lite captures sample management information electronically, the information is easily exportable to databases or various reporting formats.

Chain-of-custody forms will be signed in ink by the samplers and the individual relinquishing custody. SulTRAC will then follow the sample packaging and shipment procedures summarized below to ensure that samples arrive at the laboratory with the chain of custody intact.

1- Immediately after sample collection, sample containers will be labeled with the appropriate identifiers. Clear tape will be placed over the sample container's labels to prevent smearing.

2- The samples will be placed in Ziploc plastic bags and then in a cooler containing double-sealed bags of ice and maintained at 4 °C. The cooler will remain in a secured area or in view of the sampler until it is properly sealed for shipment to the laboratory.

3- Prior to shipping, the chain-of-custody forms, airbills, and all other relevant documents will be completed. Chain-of-custody forms will be sealed in plastic bags and taped to the inside of the cooler lid. Cushioning material, such as bubble-wrap, will be placed in the cooler.

4- A temperature blank consisting of a jar or vial containing water will be included in every cooler to be used by the laboratory to determine the cooler temperature at the time of sample receipt.

5- The shipping cooler will then be sealed with tape and custody seals in a manner that will indicate whether the cooler was opened. The preferred procedure includes placement of custody seals at diagonally opposite corners of the cooler. The custody seals will be covered with clear plastic tape or strapping tape.

The field sampler is personally responsible for the care and custody of the samples until they are transferred to other personnel or properly dispatched to an overnight carrier or directly to a laboratory. When transferring possession of the samples, the individuals relinquishing and receiving the samples sign, date, and note the time of transfer on the chain-of-custody form. Commercial carriers are not required to sign off on the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody seals remain intact.

QAPP WORKSHEET #27 (CONTINUED)
SAMPLE CUSTODY REQUIREMENTS

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): The laboratory sample custodian will receive all incoming samples and indicate receipt by signing the accompanying custody forms and retaining copies of the signed forms as permanent records. The laboratory sample custodian will record all pertinent information concerning the sample, including the persons delivering and receiving the sample, the date and time received, the method by which the sample was transmitted to the laboratory, sample condition at the time of receipt (sealed, unsealed, or broken container; temperature; or other relevant remarks), the sample identification number, and any unique laboratory identification number associated with the sample. This information should be entered into a computerized laboratory information management system (LIMS).

The laboratory will provide a secure storage area, restricted to authorized personnel, for all samples. Only the custodian can distribute samples to laboratory personnel authorized to conduct the required analyses. Laboratory analytical personnel are responsible for the care and custody of the sample upon receipt.

At the completion of sample analysis, any unused portion of the sample, together with all identifying labels, will be returned to the custodian. The returned tagged sample will be retained in secure storage until the custodian receives permission to dispose of the sample. Sample disposal will occur only on the order of the laboratory project manager in consultation with EPA or SulTRAC or when it is certain that the information is no longer required or the samples have deteriorated. Likewise, laboratory records will be maintained until the information is no longer required and final disposition is ordered by the laboratory project manager in consultation with EPA or SulTRAC.

Sample Identification Procedures: Sample identification will be as described in Section 8.2 of the FSP. Each sample will also be assigned an identifying number by CLP FORMS II Lite software. Before or during the sampling event, the user will enter information regarding the site, project, sampling team, analysis, location, matrix (SB — soil boring, BM — building material, SW — surface water, MW — monitoring well MW for groundwater), collection time and date, and sample and tag numbers.

When the laboratory receives a sample shipment, its LIMS will generate the in-house identification numbers in accordance with its sample receipt and chain-of-custody SOPs.

QAPP WORKSHEET #28
QC SAMPLES TABLE

(UFP QAPP Section 3.4)
Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil/Solid[1]
Analytical Group	VOA/CLP
Concentration Level	Low concentration
Sampling SOP	S-3, S-4, S-5, S-6
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Deuterated Monitoring Compounds	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Soil/Solid[1]
Analytical Group	SVOA/CLP
Concentration Level	Medium concentration
Sampling SOP	S-3, S-4, S-5, S-6
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Deuterated monitoring compounds	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, results are reported in narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Soil/Solid[1]
Analytical Group	PCBs/CLP
Concentration Level	Low concentration
Sampling SOP	S-3, S-4, S-5, S-6, S-18
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogate Spike	All samples	Reanalyze sample. If upon reanalysis, the surrogate meets criteria, report reanalysis results. If upon reanalysis, the surrogate does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	30-150 %R

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Soil/Solid[1]
Analytical Group	Pesticides/CLP
Concentration Level	Low concentration
Sampling SOP	S-3, S-4, S-5, S-6
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogates	All samples	Reanalyze sample. If upon reanalysis, the surrogate meets criteria, report reanalysis results. If upon reanalysis, the surrogate does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	30-150 %R

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Soil/Solid[1]
Analytical Group	TAL Metals, Mercury, and Cyanide/CLP
Concentration Level	Multi-concentration
Sampling SOP	S-3, S-4, S-5, S-6, S-7, S-16
Analytical Method/ SOP Reference	A-2, A-3, A-4, A-5
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Sensitivity/ Contamination	No target compounds > QL
MS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy/Bias	75-125 %R
Laboratory Duplicate	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Precision	<20% RPD

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Soil/Solid[1]
Analytical Group	Asbestos
Concentration Level	TBD
Sampling SOP	S-1, S-2, S-3, S-4
Analytical Method/ SOP Reference	A-9
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Laboratory Duplicate	1 per 10 samples maximum	Reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Precision	Not determined

Note:
1 Solid refers to building material sample.

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	VOA/CLP
Concentration Level	Low concentration
Sampling SOP	S-12, S-13, S-15
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias Contamination	No target compounds > QL_1
MS/MSD	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Deuterated Monitoring Compounds	All samples	Reanalyze sample. If upon reanalysis, the monitoring compound meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	SVOA/CLP
Concentration Level	Low concentration
Sampling SOP	S-12, S-13, S-15
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Deuterated monitoring compounds	All samples	Reanalyze sample. If upon reanalysis, the monitoring compounds meets criteria, report reanalysis results. If upon reanalysis, the monitoring compound does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	%R as presented in Worksheet #12

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	PCBs/CLP
Concentration Level	Low concentration
Sampling SOP	S-12, S-13, S-15
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogate Spike	All samples	Reanalyze sample. If upon reanalysis, the surrogate meets criteria, report reanalysis results. If upon reanalysis, the surrogate does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	30-150 %R

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	Pesticides/CLP
Concentration Level	Low concentration
Sampling SOP	S-12, S-13, S-15
Analytical Method/ SOP Reference	A-1
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS/MSD	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy and Precision	%R and RPD as presented in Worksheet #12
Surrogate spike	All samples	Reanalyze sample. If upon reanalysis, the surrogate meets criteria, report reanalysis results. If upon reanalysis, the surrogate does not meet criteria, the results are reported in the narrative.	Laboratory Analyst	Accuracy	30-150 %R

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	TAL Metals, Mercury, and Cyanide/CLP
Concentration Level	Multi-concentration
Sampling SOP	S-12, S-13, S-15, S-21
Analytical Method/ SOP Reference	A-2
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
MS	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Accuracy/Bias	75-125 %R
Laboratory duplicate	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Precision	<20% RPD

QAPP WORKSHEET #28 (CONTINUED)
QC SAMPLES TABLE

Matrix	Water
Analytical Group	Total Hardness
Concentration Level	Multi-concentration
Sampling SOP	S-20
Analytical Method/ SOP Reference	A-10
Sampler's Name/ Organization	Jennifer Knoepfle/SulTRAC
Analytical Organization	CLP Laboratory
No. of Sampling Locations	See Worksheet #18

QC Sample	Frequency/ Number	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. If sufficient volume is not available, reanalyze affected extracts.	Laboratory Analyst	Accuracy/Bias-Contamination	No target compounds > QL
Laboratory duplicate	1 per extraction batch of 20 samples maximum	If sufficient volume is available, extract and reanalyze samples in affected batch. Otherwise, analyze laboratory control sample to see if problem is analysis or sample.	Laboratory Analyst	Precision	<20% RPD

QAPP WORKSHEET #29
PROJECT DOCUMENTS AND RECORDS TABLE

(UFP QAPP Section 3.5.1)
Identify the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on-site and off-site analysis, and data assessment. Identify where each document will be maintained.

Document	Where Maintained
Field notes/logbook	Project file (field data), SulTRAC offices
Chain of custody forms	Project file (laboratory data), SulTRAC offices
Laboratory raw data package	EPA for CLP laboratory and subcontracted laboratory data; project file (laboratory data)
Laboratory equipment calibration logs	EPA for CLP and subcontracted laboratory
Validated data	Project file (laboratory data), SulTRAC offices

QAPP WORKSHEET #30
ANALYTICAL SERVICES TABLE

(UFP QAPP Section 3.5.2.3)
Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. Group by matrix, analytical group, concentration, and sample location or ID number. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organization cannot be used.

Matrix	Analytical Group	Concentration Level	Sampling Location/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person, and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Soil/Solid[1]	VOA	Low concentration	See Table 1, Table 2, Figure 2, and Figure 3	A-1	21 days	CLP laboratory identified by EPA Region 5	CLP laboratory identified by EPA Region 5
	SVOA	Medium concentration		A-1	21 days		
	PCBs	Low concentration		A-1	21 days		
	Pesticides	Low concentration		A-1	21 days		
	TAL Metals, Mercury, and Cyanide	Multi-concentration		A-2	21 days	Subcontacted laboratory for vegetation/soil invertebrate samples	Subcontacted laboratory for vegetation/soil invertebrate samples
	TCLP Metals	Multi-concentration		A-3	21 days		
	SPLP Metals	Multi-concentration		A-4	21 days		
	Lead/Arsenic	Multi-concentration		A-7	TBD	LEGS (Dr. Drexler)	LEGS (Dr. Drexler)
	Asbestos	Low concentration		A-8 and A-9	TBD	STAT Analysis Corporation	STAT Analysis Corporation

QAPP WORKSHEET #30 (CONTINUED)
ANALYTICAL SERVICES TABLE

Matrix	Analytical Group	Concentration Level	Sampling Location/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Water	VOA	Low concentration	See Figure 4 and Figure 5	A-1	21 days	CLP Laboratory identified by EPA Region 5	CLP Laboratory identified by EPA Region 5
	SVOA	Low concentration	See Table 2	A-1	21 days		
	PCB	Low concentration		A-1	21 days		
	Pesticide	Low concentration		A-1	21 days		
	TAL Metals, Mercury	Multi-concentration		A-2	21 days		
	Cyanide	Multi-concentration		A-2	21 days		
	Total Hardness	TBD		A-10	21 days		

Note:

1 Solids refer to building materials.

QAPP WORKSHEET #31
PLANNED PROJECT ASSESSMENTS TABLE

(UFP QAPP Section 4.1.1)
Identify the type, frequency, and responsible parties of planned assessment activities that will be performed for the project.

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organization)	Person(s) Responsible for Responding to Assessment Findings (Title and Organization)	Person(s) Responsible for Identifying and Implementing CAs (Title and Organization)	Person(s) Responsible for Monitoring Effectiveness of CAs (Title and Organization)
Off-site Laboratory Assessment	Once	Internal	SulTRAC	Richard Baldino, Project QA Manager, SulTRAC	Laboratory QA officer	TBD	TBD

QAPP WORKSHEET #32
ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

(UFP QAPP Section 4.1.2)
For each type of assessment, describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response (Name, Title, Organization)	Timeframe for Response
Off-site Laboratory Assessment	Written audit report	Laboratory QA officer	TBD	Letter	Richard Baldino, Project QA Manager, SulTRAC	TBD

QAPP WORKSHEET #33
QA MANAGEMENT REPORTS TABLE

(UFP QAPP Section 4.2)
Identify the frequency and type of planned QA Management Reports, the project delivery dates, the personnel responsible for report preparation, and the report recipients.

Type of Report	Frequency (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Name, Title, Organization)	Report Recipient(s) (Title and Organization)
Phase II Data Validation Report	Once for field sampling Phase II	21 days after receipt of Phase II analytical results from laboratory	Jennifer Knoepfle, SulTRAC, Project Manager	Demaree Collier, WAM, EPA Region 5

QAPP WORKSHEET #34
VERIFICATION (STEP I) PROCESS TABLE

(UFP QAPP Section 5.2.1)
Describe the processes that will be followed to verify project data. Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the person responsible. *Internal* or *external* is in relation to the data generator.

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain-of-custody forms	Chain-of-custody forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody form should be initialed by the reviewer, a copy of the chain-of-custody form should be retained in the project file, and the original and remaining copies should be taped inside the cooler for shipment.	Internal	Cheryl Gorman, Lea Cole, or Jennifer Knoepfle, SulTRAC
Field notes/ logbook	Field notes will be reviewed internally and placed in the project file. A copy of the field notes will be attached to the final report.	Internal	Jennifer Knoepfle, SulTRAC
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified externally in accordance with the data validation procedures specified in Worksheet #35.	Internal External	CLP Laboratory William Earle, SulTRAC

QAPP WORKSHEET #35
VALIDATION (STEPS IIA AND IIB) PROCESS TABLE

(UFP QAPP Section 5.2.2)
Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)[1]
IIa	Chain of custody	Examine traceability of samples from sample collection to sample analysis	EPA (CADRE), Analytical Coordinator, SulTRAC
IIa	Holding time	Confirm that holding time requirements are met	EPA (CADRE), Chemist, SulTRAC
IIa	Instrument calibration	Confirm that instrument calibration requirements are met	EPA (CADRE), Chemist, SulTRAC
IIa	Analytical method	Confirm that analytical methods are specified in QAPP	EPA (CADRE), Chemist, SulTRAC
IIb	Performance criteria	Confirm that QC samples meet specified performance criteria; document any deviations in data evaluation summary report	EPA (CADRE), Chemist, SulTRAC

Note:

1 EPA is responsible for conducting CADRE of analytical data generated by the CLP laboratory. SulTRAC is responsible for conducting asbestos, bioassessibility, and bioavailability validation of the analytical data generated by the subcontracted laboratory. EPA review will be conducted in accordance with CLP National Functional Guidelines (NFG) for data validation. EPA will provide SulTRAC with a summary data review report.

QAPP WORKSHEET #36
VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

(UFP QAPP Section 5.2.2)
Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Organization)
IIa	Soil/Solids	VOAs	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/ Solids	SVOAs	Medium	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/ Solids	PCBs	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/Solids	Pesticides	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/Solids	TAL Metals (mercury) and cyanide	Multi	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/Solids	TCLP Metals	Multi	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/Solids	SPLP Metals	Multi	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Soil/Solids	Asbestos	TBD	CADRE criteria and NFG	SulTRAC will validate the analytical data and review the case narrative
IIa	Vegetation	Metals	TBD	CADRE criteria and NFG	SulTRAC will validate the analytical data and review the case narrative
IIa	Soil Invertebrate Tissue	Metals	TBD	CADRE criteria and NFG	SulTRAC will validate the analytical data and review the case narrative
IIa	Soil Bioassessibility	Metals	TBD	CADRE criteria and NFG	SulTRAC will validate the analytical data and review the case narrative

QAPP WORKSHEET #36 (CONTINUED)
VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Organization)
IIa	Groundwater/Surface Water	VOAs	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Groundwater/Surface Water	SVOAs	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Groundwater/Surface Water	PCBs	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Groundwater/Surface Water	Pesticides	Low	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Groundwater/Surface Water	TAL Metals (mercury) and cyanide	Multi	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC
IIa	Surface Water	Total Hardness	TBD	CADRE criteria and NFG	CADRE validation (EPA) and review of case narrative by SulTRAC

Note:

[1] EPA is responsible for conducting CADRE of analytical data generated by the CLP laboratories. SulTRAC is responsible for conducting asbestos, bioassessibility, and bioavailability validation of the analytical data generated by the subcontracted laboratory. EPA review will be conducted in accordance with CLP NFG for data validation. EPA will provide SulTRAC with a summary data review report. The SulTRAC analytical coordinator will review this report to verify that project-specific QC criteria have been met.

QAPP WORKSHEET #37
USABILITY ASSESSMENT

(UFP QAPP Section 5.2.3)
Describe the procedures/methods/activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. Describe how data quality issues will be addressed and how limitations on the use of the data will be handled.

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: A team of SulTRAC personnel will perform the data usability assessment. SulTRAC's project manager will be responsible for information in the usability assessment. The project manager will also be responsible for assigning task work to the individual task members who will be supporting the data usability assessment. Note that the data usability assessment will be conducted on validated data. The results of the data usability assessment will be presented in the final project report.

Precision – Results of laboratory duplicates will be presented separately in tabular format. For each duplicate pair, the RPD will be calculated for each analyte whose original and duplicate values are both greater than or equal to the QL. The RPDs will be checked against the measurement performance criteria presented in Worksheet #12. The RPDs exceeding criteria will be identified in the tables. Additionally, the RPD of each analyte will be averaged across all duplicate pairs whose original and duplicate values are both greater than or equal to the QL, and the combined overall average RPD for each analysis will be calculated for the laboratory duplicates. A discussion will follow summarizing the laboratory precision results. Any conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias – Results for laboratory method blanks and instrument blanks will be presented separately in tabular format for each analysis for both Aroclors/pesticides and mercury. The results for each analyte will be checked against the measurement performance criteria presented in Worksheet #12. Results for analytes that exceed criteria will be identified in the tables. A discussion will follow summarizing the laboratory accuracy/bias results. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn, and any limitations on the use of the data will be described.

Overall Accuracy/Bias – The results will be presented in tabular format to allow comparison of these results to the sample batch they apply to. These results will be compared to the requirements listed in Worksheet #12. A discussion will follow summarizing overall accuracy/bias results. Any conclusions about the overall accuracy/bias of the analyses will be drawn, and any limitations on the use of the data will be described.

Sensitivity – Results for all laboratory-fortified blanks will be presented separately in tabular format for each analysis. The results for each analyte will be checked against the measurement performance criteria presented in Worksheet #12 and cross-checked against the QLs presented in Worksheet #15. Results for analytes that exceed criteria will be identified on the tables. A discussion will follow summarizing the laboratory sensitivity results. Any conclusions about the sensitivity of the analyses will be drawn, and any limitations on the use of the data will be described.

Representativeness – The large numbers of samples collected in Phases I and II are considered representative of site conditions, as long as completeness criteria in Worksheet #12 are met.

QAPP WORKSHEET #37 (CONTINUED)
USABILITY ASSESSMENT

Comparability – The results of this study will be used as a benchmark for determining comparability for data collected during any potential future sampling events using the same or similar sampling and analytical SOPs. **Completeness** – A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented in Worksheet #12. Completeness will be calculated for each analyte as follows. For each analyte, completeness will be calculated as the number of data points for each analyte and individual matrix that meet the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each analyte. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn, and any limitations on the use of the data will be described.
Describe the evaluative procedures used to assess overall measurement error associated with the project: project: NA
Identify the personnel responsible for performing the usability assessment: SulTRAC's analytical coordinator will review analytical data and the CADRE data review report to assess usability of the data. SulTRAC's project manager will review RPDs for samples and assess the overall usability of the data set in close consultation with the EPA WAM.
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The usability assessment will be documented in the data validation letter report, which will be generated 21 days after Phase II analytical results are received from the CLP laboratory.

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TABLES

(11 Sheets)

TABLE 1
SOIL BORING LOCATIONS
Matthiessen and Hegeler Zinc Company Site,
Operable Unit 2

Building Number	Location	Use and Environmental Concern	Number of Soil Borings	Number of Samples
None	North of main industrial area	Vegetated area where slag, sinter, and debris formerly dumped	10	20
None	Northeast periphery	Natural area recently found to contain three outflow pipes with water flowing	10	20
Multiple buildings	Main industrial area	Heavy metals found during Phase I; data gap investigation for Phase II	10	20
100	Building 100 area	High PCB concentrations found during Phase I; three tracks for locomotive repair work	10	20
1-2-3-4-105-117	Northwest corner of the rolling mill	TCE found during Phase I	10	20
1-2-3-4-105-117	Rolling mill (inside)	Rolling zinc, coal fuel, oil-filled transformers, reheated zinc, heavy oil on floor, power house	10	20

Notes:

TCE Trichloroethene
OU Operable unit
PCB Polychlorinated biphenyl

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB401A-08	Soil boring	Field	SB401	Near Building 100	0-2				x	x											
SB401B-08	Soil boring	Field	SB401	Near Building 100	>12				x	x								x			
SB402A-08	Soil boring	Field	SB402	Near Building 100	0-2				x	x											
SB402B-08	Soil boring	Field	SB402	Near Building 100	>12				x	x								x			
SB403A-08	Soil boring	Field	SB403	Near Building 100	0-2				x	x											
SB403B-08	Soil boring	Field	SB403	Near Building 100	>12				x	x								x			
SB404A-08	Soil boring	Field	SB404	Near Building 100	0-2				x	x											
SB404B-08	Soil boring	Field	SB404	Near Building 100	>12				x	x								x			
SB405A-08	Soil boring	Field	SB405	Near Building 100	0-2				x	x											
SB405B-08	Soil boring	Field	SB405	Near Building 100	>12				x	x								x			
SB406A-08	Soil boring	Field	SB406	Near Building 100	0-2				x	x						x					
SB406B-08	Soil boring	Field	SB406	Near Building 100	>12				x	x						x		x			
SB407A-08	Soil boring	Field	SB407	Near Building 100	0-2				x	x											
SB407B-08	Soil boring	Field	SB407	Near Building 100	>12				x	x								x			
SB408A-08	Soil boring	Field	SB408	Near Building 100	0-2				x	x											
SB408B-08	Soil boring	Field	SB408	Near Building 100	>12				x	x								x			
SB409A-08	Soil boring	Field	SB409	Near Building 100	0-2				x	x											
SB409B-08	Soil boring	Field	SB409	Near Building 100	>12				x	x								x			
SB410A-08	Soil boring	Field	SB410	Near Building 100	0-2				x	x							x				
SB410B-08	Soil boring	Field	SB410	Near Building 100	>12				x	x								x			
SB411A-08	Soil boring	Field	SB411	NW of Rolling Mill	0-2	x				x											
SB411B-08	Soil boring	Field	SB411	NW of Rolling Mill	2-12	x				x											
SB412A-08	Soil boring	Field	SB412	NW of Rolling Mill	0-2	x				x						x					
SB412B-08	Soil boring	Field	SB412	NW of Rolling Mill	2-12	x				x						x					
SB413A-08	Soil boring	Field	SB413	NW of Rolling Mill	0-2	x				x											
SB413B-08	Soil boring	Field	SB413	NW of Rolling Mill	2-12	x				x											
SB414A-08	Soil boring	Field	SB414	NW of Rolling Mill	0-2	x				x											
SB414B-08	Soil boring	Field	SB414	NW of Rolling Mill	2-12	x				x											
SB415A-08	Soil boring	Field	SB415	NW of Rolling Mill	0-2	x				x											
SB415B-08	Soil boring	Field	SB415	NW of Rolling Mill	2-12	x				x											
SB416A-08	Soil boring	Field	SB416	NW of Rolling Mill	0-2	x				x								x			
SB416B-08	Soil boring	Field	SB416	NW of Rolling Mill	2-12	x				x											
SB417A-08	Soil boring	Field	SB417	NW of Rolling Mill	0-2	x				x											
SB417B-08	Soil boring	Field	SB417	NW of Rolling Mill	2-12	x				x											
SB418A-08	Soil boring	Field	SB418	NW of Rolling Mill	0-2	x				x											
SB418B-08	Soil boring	Field	SB418	NW of Rolling Mill	2-12	x				x											
SB419A-08	Soil boring	Field	SB419	NW of Rolling Mill	0-2	x				x											
SB419B-08	Soil boring	Field	SB419	NW of Rolling Mill	2-12	x				x											
SB420A-08	Soil boring	Field	SB420	NW of Rolling Mill	0-2	x				x											
SB420B-08	Soil boring	Field	SB420	NW of Rolling Mill	2-12	x				x											
SB421A-08	Soil boring	Field	SB421	North Area	0-2	x	x	x	x	x				x							
SB421B-08	Soil boring	Field	SB421	North Area	2-12	x	x	x	x	x											
SB422A-08	Soil boring	Field	SB422	North Area	0-2	x	x	x	x	x				x							
SB422B-08	Soil boring	Field	SB422	North Area	2-12	x	x	x	x	x											
SB423A-08	Soil boring	Field	SB423	North Area	0-2	x	x	x	x	x				x		x					
SB423B-08	Soil boring	Field	SB423	North Area	2-12	x	x	x	x	x						x					
SB424A-08	Soil boring	Field	SB424	North Area	0-2	x	x	x	x	x				x							
SB424B-08	Soil boring	Field	SB424	North Area	2-12	x	x	x	x	x											
SB425A-08	Soil boring	Field	SB425	North Area	0-2	x	x	x	x	x				x							
SB425B-08	Soil boring	Field	SB425	North Area	2-12	x	x	x	x	x											
SB426A-08	Soil boring	Field	SB426	North Area	0-2	x	x	x	x	x				x							
SB426B-08	Soil boring	Field	SB426	North Area	2-12	x	x	x	x	x											
SB427A-08	Soil boring	Field	SB427	North Area	0-2	x	x	x	x	x				x				x			
SB427B-08	Soil boring	Field	SB427	North Area	2-12	x	x	x	x	x											
SB428A-08	Soil boring	Field	SB428	North Area	0-2	x	x	x	x	x				x							
SB428B-08	Soil boring	Field	SB428	North Area	2-12	x	x	x	x	x											
SB429A-08	Soil boring	Field	SB429	North Area	0-2	x	x	x	x	x				x							
SB429B-08	Soil boring	Field	SB429	North Area	2-12	x	x	x	x	x											
SB430A-08	Soil boring	Field	SB430	North Area	0-2	x	x	x	x	x				x							
SB430B-08	Soil boring	Field	SB430	North Area	2-12	x	x	x	x	x											
SB431A-08	Soil boring	Field	SB431	Inside Rolling Mill	0-2	x	x	x	x	x				x				x			
SB431B-08	Soil boring	Field	SB421	Inside Rolling Mill	2-12	x	x	x	x	x											
SB432A-08	Soil boring	Field	SB432	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB432B-08	Soil boring	Field	SB432	Inside Rolling Mill	2-12	x	x	x	x	x											
SB433A-08	Soil boring	Field	SB433	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB433B-08	Soil boring	Field	SB433	Inside Rolling Mill	2-12	x	x	x	x	x											
SB434A-08	Soil boring	Field	SB434	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB434B-08	Soil boring	Field	SB434	Inside Rolling Mill	2-12	x	x	x	x	x											

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB435A-08	Soil boring	Field	SB435	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB435B-08	Soil boring	Field	SB435	Inside Rolling Mill	2-12	x	x	x	x	x											
SB436A-08	Soil boring	Field	SB436	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB436B-08	Soil boring	Field	SB436	Inside Rolling Mill	2-12	x	x	x	x	x											
SB437A-08	Soil boring	Field	SB437	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB437B-08	Soil boring	Field	SB437	Inside Rolling Mill	2-12	x	x	x	x	x											
SB438A-08	Soil boring	Field	SB438	Inside Rolling Mill	0-2	x	x	x	x	x				x		x					
SB438B-08	Soil boring	Field	SB438	Inside Rolling Mill	2-12	x	x	x	x	x						x					
SB439A-08	Soil boring	Field	SB439	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB439B-08	Soil boring	Field	SB439	Inside Rolling Mill	2-12	x	x	x	x	x											
SB440A-08	Soil boring	Field	SB440	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB440B-08	Soil boring	Field	SB440	Inside Rolling Mill	2-12	x	x	x	x	x											
SB441A-08	Soil boring	Field	SB441	NE Periphery Area	0-2	x	x	x	x	x				x							
SB441B-08	Soil boring	Field	SB441	NE Periphery Area	2-12	x	x	x	x	x											
SB442A-08	Soil boring	Field	SB442	NE Periphery Area	0-2	x	x	x	x	x				x							
SB442B-08	Soil boring	Field	SB442	NE Periphery Area	2-12	x	x	x	x	x											
SB443A-08	Soil boring	Field	SB443	NE Periphery Area	0-2	x	x	x	x	x				x							
SB443B-08	Soil boring	Field	SB443	NE Periphery Area	2-12	x	x	x	x	x											
SB444A-08	Soil boring	Field	SB444	NE Periphery Area	0-2	x	x	x	x	x				x		x					
SB444B-08	Soil boring	Field	SB444	NE Periphery Area	2-12	x	x	x	x	x						x					
SB445A-08	Soil boring	Field	SB445	NE Periphery Area	0-2	x	x	x	x	x				x							
SB445B-08	Soil boring	Field	SB445	NE Periphery Area	2-12	x	x	x	x	x											
SB446A-08	Soil boring	Field	SB446	NE Periphery Area	0-2	x	x	x	x	x				x							
SB446B-08	Soil boring	Field	SB446	NE Periphery Area	2-12	x	x	x	x	x											
SB447A-08	Soil boring	Field	SB447	NE Periphery Area	0-2	x	x	x	x	x				x							
SB447B-08	Soil boring	Field	SB447	NE Periphery Area	2-12	x	x	x	x	x											
SB448A-08	Soil boring	Field	SB448	NE Periphery Area	0-2	x	x	x	x	x				x							
SB448B-08	Soil boring	Field	SB448	NE Periphery Area	2-12	x	x	x	x	x											
SB449A-08	Soil boring	Field	SB449	NE Periphery Area	0-2	x	x	x	x	x				x							
SB449B-08	Soil boring	Field	SB449	NE Periphery Area	2-12	x	x	x	x	x											
SB450A-08	Soil boring	Field	SB450	NE Periphery Area	0-2	x	x	x	x	x				x			x				
SB450B-08	Soil boring	Field	SB450	NE Periphery Area	2-12	x	x	x	x	x											
SB451A-08	Soil boring	Field	SB451	Main Industrial Area	0-2					x		x	x								
SB451B-08	Soil boring	Field	SB451	Main Industrial Area	2-12					x		x	x								
SB452A-08	Soil boring	Field	SB452	Main Industrial Area	0-2					x		x	x			x					
SB452B-08	Soil boring	Field	SB452	Main Industrial Area	2-12					x		x	x								
SB453A-08	Soil boring	Field	SB453	Main Industrial Area	0-2					x		x	x								
SB453B-08	Soil boring	Field	SB453	Main Industrial Area	2-12					x		x	x								
SB454A-08	Soil boring	Field	SB454	Main Industrial Area	0-2					x		x	x								
SB454B-08	Soil boring	Field	SB454	Main Industrial Area	2-12					x		x	x				x				
SB455A-08	Soil boring	Field	SB455	Main Industrial Area	0-2					x		x	x								
SB455B-08	Soil boring	Field	SB455	Main Industrial Area	2-12					x		x	x								
SB456A-08	Soil boring	Field	SB456	Main Industrial Area	0-2					x			x								
SB456B-08	Soil boring	Field	SB456	Main Industrial Area	2-12					x			x								
SB457A-08	Soil boring	Field	SB457	Main Industrial Area	0-2					x			x			x					
SB457B-08	Soil boring	Field	SB457	Main Industrial Area	2-12					x			x								
SB458A-08	Soil boring	Field	SB458	Main Industrial Area	0-2					x			x								
SB458B-08	Soil boring	Field	SB458	Main Industrial Area	2-12					x			x								
SB459A-08	Soil boring	Field	SB459	Main Industrial Area	0-2					x			x								
SB459B-08	Soil boring	Field	SB459	Main Industrial Area	2-12					x			x								
SB460A-08	Soil boring	Field	SB460	Main Industrial Area	0-2					x			x								
SB460B-08	Soil boring	Field	SB460	Main Industrial Area	2-12					x			x								
SB500A-08	Soil boring	Duplicate	SB406A	Near Building 100	0-2				x	x											
SB500B-08	Soil boring	Duplicate	SB406B	Near Building 100	2-12				x	x											
SB501A-08	Soil boring	Duplicate	SB412A	NW of Rolling Mill	0-2	x				x											
SB501B-08	Soil boring	Duplicate	SB412A	NW of Rolling Mill	2-12	x				x											
SB502A-08	Soil boring	Duplicate	SB423A	North Area	0-2	x	x	x	x	x				x							
SB502B-08	Soil boring	Duplicate	SB423B	North Area	2-12	x	x	x	x	x											
SB503A-08	Soil boring	Duplicate	SB438A	Inside Rolling Mill	0-2	x	x	x	x	x				x							
SB503B-08	Soil boring	Duplicate	SB438B	Inside Rolling Mill	2-12	x	x	x	x	x											
SB504A-08	Soil boring	Duplicate	SB444A	NE Periphery Area	0-2	x	x	x	x	x				x							
SB504B-08	Soil boring	Duplicate	SB444B	NE Periphery Area	2-12	x	x	x	x	x											
SB505A-08	Soil boring	Duplicate	SB452A	Main Industrial Area	0-2					x		x	x								
SB505B-08	Soil boring	Duplicate	SB457A	Main Industrial Area	0-2					x		x	x								
SB506-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB507-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB508-08	Soil boring	Duplicate	TBD	TBD	TBD																
SB509-08	Soil boring	Duplicate	TBD	TBD	TBD																

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SB510R-08	Soil boring	Rinsate	NA	Week 1	NA	x	x	x	x	x											
SB511R-08	Soil boring	Rinsate	NA	Week 2	NA	x	x	x	x	x											
SB512R-08	Soil boring	Rinsate	NA	Week 3	NA	x	x	x	x	x											
SB513R-08	Soil boring	Rinsate	NA	Week 4	NA	x	x	x	x	x											
SB514R-08	Soil boring	Rinsate	NA	Week 5	NA	x	x	x	x	x											
poly_1_001	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_002	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_003	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_004	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_005	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_006	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_007	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_008	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_009	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_010	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_011	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_012	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_013	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_014	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_015	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_016	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_017	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_018	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_019	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_020	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_021	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_022	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_023	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_024	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_025	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_026	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_027	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_028	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_029	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_030	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_031	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_032	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_033	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_034	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_035	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_036	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_037	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_038	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_039	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_040	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_041	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_042	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_043	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_044	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_045	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_046	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_047	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_048	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_049	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_050	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_051	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_052	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_053	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_054	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_055	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_056	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_057	Surface Soil	Field	poly_1[2]	North Area	0-1																ns

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_1_058	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_059	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_060	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_061	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_062	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_063	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_064	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_065	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_066	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_067	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_068	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_069	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_070	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_071	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_072	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_073	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_074	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_075	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_076	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_077	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_078	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_079	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_080	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_081	Surface Soil	Field	poly_1[2]	North Area	0-1																ns
poly_1_082	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_083	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_084	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_085	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_086	Surface Soil	Field	poly_1[2]	North Area	0-1					x											x
poly_1_087	Surface Soil	Field	poly_1[2]	North Area	0-1					x							x				x
poly_1_088	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_089	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_1_090	Surface Soil	Field	poly_1[2]	North Area	0-1					x						x					x
poly_1_091	Surface Soil	Field	poly_1[2]	North Area	0-1																x
poly_2_001	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_002	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_003	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_004	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_005	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_006	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_007	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_008	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_009	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_010	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_011	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_012	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_013	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_014	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_015	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_016	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_017	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_018	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_019	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_020	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_021	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_022	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_023	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_024	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_025	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_026	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_027	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_2_028	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_029	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_030	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_031	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_032	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_033	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_034	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_035	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_036	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_037	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x						x					x
poly_2_038	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_039	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x							x				x
poly_2_040	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_041	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_042	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_043	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_044	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_045	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_046	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_047	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_048	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_049	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_050	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_051	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_052	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_053	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_054	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_055	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x						x					x
poly_2_056	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_057	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_058	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_059	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_060	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_061	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_062	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_063	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_064	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_065	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_066	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_067	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_068	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_069	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_070	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_071	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_072	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_073	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_074	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_075	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_076	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_077	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_078	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_079	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_080	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_081	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_082	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_083	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_084	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_085	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1					x											x
poly_2_086	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_087	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_2_088	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_2_089	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_090	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																ns
poly_2_091	Surface Soil	Field	poly_2[2]	NE Periphery Area	0-1																x
poly_3_001	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_002	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																ns
poly_3_003	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_004	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_005	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_006	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_007	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_008	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_009	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x						x					x
poly_3_010	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_011	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_012	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_013	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_3_014	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1																x
poly_3_015	Surface Soil	Field	poly_3[2]	Main Industrial Area - Central Eastern Area	0-1					x											x
poly_4_001	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_002	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_003	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_004	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_005	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_006	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_007	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_008	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x						x					x
poly_4_009	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_010	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_011	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1					x											x
poly_4_012	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_4_013	Surface Soil	Field	poly_4[2]	Main Industrial Area - Southeastern Area	0-1																x
poly_5_001	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x											x
poly_5_002	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_003	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_004	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_005	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_006	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_007	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_008	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x							x				x
poly_5_009	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_010	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_011	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_012	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_013	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_014	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_015	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_016	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_017	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_018	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_019	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1					x											x
poly_5_020	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_021	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_022	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_023	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																x
poly_5_024	Surface Soil	Field	poly_5[2]	Main Industrial Area - Western Area	0-1																ns
poly_6_001	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																ns
poly_6_002	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_003	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_004	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_005	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_006	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
poly_6_007	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_008	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_6_009	Surface Soil	Field	poly_6[2]	Main Industrial Area - Southwestern Area	0-1																x
poly_1_200	Surface Soil	Duplicate	poly_1[2]	Duplicate of poly_1_090	0-1					x											x
poly_2_200	Surface Soil	Duplicate	poly_2[2]	Duplicate of poly_2_037	0-1					x											x
poly_2_201	Surface Soil	Duplicate	poly_2[2]	Duplicate of poly_2_055	0-1					x											x
poly_3_200	Surface Soil	Duplicate	poly_3[2]	Duplicate of poly_3_009	0-1					x											x
poly_4_200	Surface Soil	Duplicate	poly_4[2]	Duplicate of poly_4_008	0-1					x											x
SS001-08	Surface Soil	Field	SS001	Main Industrial Area (Area A)	0-2														x		
SS002-08	Surface Soil	Field	SS002	Main Industrial Area (Area A)	0-2														x		
SS003-08	Surface Soil	Field	SS003	Main Industrial Area (Area A)	0-2														x		
SS004-08	Surface Soil	Field	SS004	North Area (Area B)	0-2														x		
SS005-08	Surface Soil	Field	SS005	North Area (Area B)	0-2														x		
SS006-08	Surface Soil	Field	SS006	NE Periphery Area (Area C)	0-2														x		
SS007-08	Surface Soil	Field	SS007	NE Periphery Area (Area C)	0-2														x		
SS008-08	Surface Soil	Field	SS008	Near Building 100 (Area D)	0-2														x		
SS009-08	Surface Soil	Field	SS009	NW of Rolling Mill (Area E)	0-2														x		
SS010-08	Surface Soil	Field	SS010	Residential Area (Area F)	0-2														x		
SS011-08	Surface Soil	Field	SS011	Area East of River (Area G)	0-2														x		
SS012-08	Surface Soil	Field	SS012	Disturbed Woodland-Grassland	0-2															x	
SS013-08	Surface Soil	Field	SS013	Disturbed Woodland-Grassland	0-2															x	
SS014-08	Surface Soil	Field	SS014	Disturbed Woodland-Grassland	0-2															x	
SS015-08	Surface Soil	Field	SS015	Disturbed Woodland-Grassland	0-2															x	
SS016-08	Surface Soil	Field	SS016	Disturbed Woodland-Grassland	0-2															x	
SS017-08	Surface Soil	Field	SS017	Disturbed Woodland-Grassland	0-2															x	
SS018-08	Surface Soil	Field	SS018	Disturbed Woodland-Grassland	0-2															x	
SS019-08	Surface Soil	Field	SS019	Disturbed Woodland-Grassland	0-2															x	
SS020-08	Surface Soil	Field	SS020	Disturbed Woodland-Grassland	0-2															x	
SS021-08	Surface Soil	Field	SS021	Disturbed Woodland-Grassland	0-2															x	
SS022-08	Surface Soil	Field	SS022	Oak-Hickory Woodland	0-2															x	
SS023-08	Surface Soil	Field	SS023	Oak-Hickory Woodland	0-2															x	
SS024-08	Surface Soil	Field	SS024	Oak-Hickory Woodland	0-2															x	
SS025-08	Surface Soil	Field	SS025	Oak-Hickory Woodland	0-2															x	
SS026-08	Surface Soil	Field	SS026	Oak-Hickory Woodland	0-2															x	
SS027-08	Surface Soil	Field	SS027	Oak-Hickory Woodland	0-2															x	
SS028-08	Surface Soil	Field	SS028	Oak-Hickory Woodland	0-2															x	
SS029-08	Surface Soil	Field	SS029	Oak-Hickory Woodland	0-2															x	
SS030-08	Surface Soil	Field	SS030	Oak-Hickory Woodland	0-2															x	
SS031-08	Surface Soil	Field	SS031	Oak-Hickory Woodland	0-2															x	
SS032-08	Surface Soil	Field	SS032	Savannah	0-2															x	
SS033-08	Surface Soil	Field	SS033	Savannah	0-2															x	
SS034-08	Surface Soil	Field	SS034	Savannah	0-2															x	
SS035-08	Surface Soil	Field	SS035	Savannah	0-2															x	
SS036-08	Surface Soil	Field	SS036	Savannah	0-2															x	
SS037-08	Surface Soil	Field	SS037	Savannah	0-2															x	
SS038-08	Surface Soil	Field	SS038	Savannah	0-2															x	
SS039-08	Surface Soil	Field	SS039	Savannah	0-2															x	
SS040-08	Surface Soil	Field	SS040	Savannah	0-2															x	
SS041-08	Surface Soil	Field	SS041	Savannah	0-2															x	
SS042-08	Surface Soil	Field	SS042	Riverine	0-2															x	
SS043-08	Surface Soil	Field	SS043	Riverine	0-2															x	
SS044-08	Surface Soil	Field	SS044	Riverine	0-2															x	
SS045-08	Surface Soil	Field	SS045	Riverine	0-2															x	
SS046-08	Surface Soil	Field	SS046	Riverine	0-2															x	
SS047-08	Surface Soil	Field	SS047	Riverine	0-2															x	
SS048-08	Surface Soil	Field	SS048	Riverine	0-2															x	
SS049-08	Surface Soil	Field	SS049	Riverine	0-2															x	
SS050-08	Surface Soil	Field	SS050	Riverine	0-2															x	
SS051-08	Surface Soil	Field	SS051	Riverine	0-2															x	
SS052-08	Surface Soil	Field	SS052	Vegetation above ground	0-2															x	
SS053-08	Surface Soil	Field	SS053	Vegetation below ground	0-2															x	
SS054-08	Surface Soil	Field	SS054	Vegetation above ground	0-2															x	
SS055-08	Surface Soil	Field	SS055	Vegetation below ground	0-2															x	
SS056-08	Surface Soil	Field	SS056	Vegetation above ground	0-2															x	
SS057-08	Surface Soil	Field	SS057	Vegetation below ground	0-2															x	
SS058-08	Surface Soil	Field	SS058	Vegetation above ground	0-2															x	
SS059-08	Surface Soil	Field	SS059	Vegetation below ground	0-2															x	

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
SS060-08	Surface Soil	Field	SS060	Vegetation above ground	0-2															x	
SS061-08	Surface Soil	Field	SS061	Vegetation below ground	0-2															x	
SS062-08	Surface Soil	Field	SS062	Vegetation above ground	0-2															x	
SS063-08	Surface Soil	Field	SS063	Vegetation below ground	0-2															x	
SS100-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS101-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS102-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS103-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS104-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS105-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
SS106-08	Surface Soil	Duplicate	TBD	TBD by Ecologist in Field	0-2																
BM011K-08	Building Material[3,4]	Field	BM011	Ore Storage Building	NA		x	x	x	x				x							
BM012T-08	Building Material[3,4]	Field	BM012	Pottery Building	NA		x	x	x	x				x		x					
BM013Z-08	Building Material[3,4]	Field	BM013	Acid Reservoir 3	NA		x	x	x	x		x		x							
BM014T-08	Building Material[3,4]	Field	BM014	Acid Reservoir 4	NA		x	x	x	x				x							
BM015Z-08	Building Material[3,4]	Field	BM015	System 1&2	NA		x	x	x	x				x		x					
BM016C-08	Building Material[3,4]	Field	BM016	System 1&2	NA		x	x	x	x				x							
BM017C-08	Building Material[3,4]	Field	BM017	System 4	NA		x	x	x	x				x							
BM018K-08	Building Material[3,4]	Field	BM018	Refining Plant	NA		x	x	x	x				x							
BM019C-08	Building Material[3,4]	Field	BM019	Refining Plant	NA		x	x	x	x				x							
BM020K-08	Building Material[3,4]	Field	BM020	Building 100	NA		x	x	x	x				x							
BM021C-08	Building Material[3,4]	Field	BM021	Building 100	NA		x	x	x	x				x			x				
BM022K-08	Building Material[3,4]	Field	BM022	Locomotive Building (PCB Area)	NA		x	x	x	x				x							
BM023K-08	Building Material[3,4]	Field	BM023	System 5	NA		x	x	x	x				x							
BM024C-08	Building Material[3,4]	Field	BM024	System 5	NA		x	x	x	x				x							
BM025W-08	Building Material[3,4]	Field	BM025	Ore Storage	NA	x	x	x	x	x				x							
BM026K-08	Building Material[3,4]	Field	BM026	Ore Storage	NA		x	x	x	x				x							
BM027Z-08	Building Material[3,4]	Field	BM027	System 4	NA		x	x	x	x				x							
BM028C-08	Building Material[3,4]	Field	BM028	System 4/Kiln	NA		x	x	x	x		x		x							
BM029W-08	Building Material[3,4]	Field	BM029	System 3	NA	x	x	x	x	x				x							
BM030C-08	Building Material[3,4]	Field	BM030	Coke Crushing	NA		x	x	x	x				x		x					
BM031K-08	Building Material[3,4]	Field	BM031	Oxide Plant	NA		x	x	x	x				x							
BM032K-08	Building Material[3,4]	Field	BM032	Oxide Plant	NA		x	x	x	x				x							
BM033Z-08	Building Material[3,4]	Field	BM033	Tile Pile West of Acid Tanks	NA		x	x	x	x				x							
BM034T-08	Building Material[3,4]	Field	BM034	Top of Furnaces	NA		x	x	x	x				x							
BM035C-08	Building Material[3,4]	Field	BM035	Concentration Plant	NA		x	x	x	x				x							
BM036Z-08	Building Material[3,4]	Field	BM036	Building 101	NA		x	x	x	x				x							
BM037K-08	Building Material[3,4]	Field	BM037	Building 101	NA		x	x	x	x				x							
BM038C-08	Building Material[3,4]	Field	BM038	Top of Furnaces	NA		x	x	x	x				x							
BM039K-08	Building Material[3,4]	Field	BM039	Furnaces	NA		x	x	x	x				x		x					
BM040K-08	Building Material[3,4]	Field	BM040	South of System 4/Kilns/Outfall	NA		x	x	x	x				x							
BM041C-08	Building Material[3,4]	Field	BM041	Sintering Plant	NA		x	x	x	x				x							
BM042W-08	Building Material[3,4]	Field	BM042	Hoisting Engineer	NA	x	x	x	x	x		x		x							
BM043K-08	Building Material[3,4]	Field	BM043	Building 1943	NA		x	x	x	x				x							
BM044C-08	Building Material[3,4]	Field	BM044	Building 1943	NA		x	x	x	x				x							
BM045K-08	Building Material[3,4]	Field	BM045	Rolling Mill	NA		x	x	x	x				x			x				
BM046W-08	Building Material[3,4]	Field	BM046	Boiler House	NA	x	x	x	x	x				x							
BM047Z-08	Building Material[3,4]	Field	BM047	Rolling Mill	NA		x	x	x	x				x							
BM048C-08	Building Material[3,4]	Field	BM048	Rolling Mill	NA		x	x	x	x				x							
BM049K-08	Building Material[3,4]	Field	BM049	Office Building	NA		x	x	x	x		x		x							
BM050K-08	Building Material[3,4]	Field	BM050	Brick Pile East of Acid Reservoir 8	NA		x	x	x	x				x							
BM051C-08	Building Material[3,4]	Field	BM051	Acid Tank	NA		x	x	x	x				x							
BM052C-08	Building Material[3,4]	Field	BM052	Acid Tank	NA		x	x	x	x				x		x					
BM053K-08	Building Material[3,4]	Field	BM053	Top of Furnaces	NA		x	x	x	x				x							
BM054C-08	Building Material[3,4]	Field	BM054	Acid Tank	NA		x	x	x	x				x							
BM055C-08	Building Material[3,4]	Field	BM055	Acid Reservoir 9	NA		x	x	x	x				x							
BM056Z-08	Building Material[3,4]	Field	BM056	Acid Reservoir 7	NA		x	x	x	x				x							
BM057K-08	Building Material[3,4]	Field	BM057	Acid Reservoir 6	NA		x	x	x	x				x							
BM058W-08	Building Material[3,4]	Field	BM058	Wood Pile North of Acid Tanks	NA	x	x	x	x	x				x			x				
BM059K-08	Building Material[3,4]	Field	BM059	Brick Pile East of Acid Reservoir 8	NA		x	x	x	x		x		x							
BM060Z-08	Building Material[3,4]	Field	BM060	Pump House	NA		x	x	x	x				x							
BM200T-08	Building Material[3,4]	Duplicate	BM012	Pottery Building	NA		x	x	x	x				x							

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered: Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
BM201Z-08	Building Material[3,4]	Duplicate	BM015	System 1&2	NA		x	x	x	x				x							
BM202C-08	Building Material[3,4]	Duplicate	BM030	System 3 - Coke Crushing	NA		x	x	x	x				x							
BM203K-08	Building Material[3,4]	Duplicate	BM039	Furnaces	NA		x	x	x	x				x							
BM204C-08	Building Material[3,4]	Duplicate	BM052	Acid Tank	NA		x	x	x	x				x							
MW01-mm08	Groundwater[5]	Field	MW-01	Western Border		x	x	x	x	x							x				
MW02S-mm08	Groundwater[5]	Field	MW-02S	Western Border		x	x	x	x	x											
MW02D-mm08	Groundwater[5]	Field	MW-02D	Western Border		x	x	x	x	x											
MW03-mm08	Groundwater[5]	Field	MW-03	Western Border		x	x	x	x	x											
MW04-mm08	Groundwater[5]	Field	MW-04	Northeast of Rolling Mill		x	x	x	x	x											
MW05-mm08	Groundwater[5]	Field	MW-05	West of Furnaces		x	x	x	x	x						x					
MW06-mm08	Groundwater[5]	Field	MW-06	Main Industrial Area		x	x	x	x	x											
MW07-mm08	Groundwater[5]	Field	MW-07	North of Furnaces		x	x	x	x	x											
MW08-mm08	Groundwater[5]	Field	MW-08	South of Furnaces		x	x	x	x	x											
MW09-mm08	Groundwater[5]	Field	MW-09	Main Industrial Area		x	x	x	x	x											
MW10-mm08	Groundwater[5]	Field	MW-10	North of Building 100		x	x	x	x	x											
MW11-mm08	Groundwater[5]	Field	MW-11	Main Industrial Area		x	x	x	x	x											
MW12-mm08	Groundwater[5]	Field	MW-12	South of Acid Reservoirs		x	x	x	x	x											
MW13-mm08	Groundwater[5]	Field	MW-13	North of Pottery Building		x	x	x	x	x											
MW14-mm08	Groundwater[5]	Field	MW-14	North of Acid Reservoirs		x	x	x	x	x							x				
MW15-mm08	Groundwater[5]	Field	MW-15	North Area		x	x	x	x	x						x					
MW16-mm08	Groundwater[5]	Field	MW-16	North Area		x	x	x	x	x											
MW17-mm08	Groundwater[5]	Field	MW-17	Main Industrial Area		x	x	x	x	x						x					
MW18-mm08	Groundwater[5]	Field	MW-18	Next to Little Vermillion River		x	x	x	x	x						x					
MW19-mm08	Groundwater[5]	Field	MW-19	East of Main Industrial Area		x	x	x	x	x											
MW20-mm08	Groundwater[5]	Field	MW-20	East of Main Industrial Area		x	x	x	x	x											
MW21-mm08	Groundwater[5]	Field	MW-21	Next to Pump House		x	x	x	x	x											
MW22-mm08	Groundwater[5]	Field	MW-22	North Area		x	x	x	x	x											
MW23-mm08	Groundwater[5]	Field	MW-23	Main Industrial Area		x	x	x	x	x											
MW24-mm08	Groundwater[5]	Field	MW-24	Main Industrial Area		x	x	x	x	x											
MW25-mm08	Groundwater[5]	Field	MW-25	Main Industrial Area		x	x	x	x	x											
MW26-mm08	Groundwater[5]	Field	MW-26	Western Border		x	x	x	x	x											
MW27-mm08	Groundwater[5]	Field	MW-27	Building 100		x	x	x	x	x											
MW28-mm08	Groundwater[5]	Field	MW-28	East of Building 100		x	x	x	x	x											
MW29-mm08	Groundwater[5]	Field	MW-29	Western Border near Rolling Mill		x	x	x	x	x											
MW30-mm08	Groundwater[5]	Field	MW-30	North of Rolling Mill		x	x	x	x	x											
MW31-mm08	Groundwater[5]	Field	MW-31	North of Rolling Mill		x	x	x	x	x											
MW32-mm08	Groundwater[5]	Field	MW-32	East of Rolling Mill		x	x	x	x	x											
MW33-mm08	Groundwater[5]	Field	MW-33	East of Rolling Mill		x	x	x	x	x											
MW34-mm08	Groundwater[5]	Field	MW-34	South of Rolling Mill		x	x	x	x	x											
MW35-mm08	Groundwater[5]	Field	MW-35	North Area		x	x	x	x	x											
MW50-mm08	Groundwater[5]	Duplicate	MW-15	North Area		x	x	x	x	x											
MW51-mm08	Groundwater[5]	Duplicate	MW-17	Main Industrial Area		x	x	x	x	x											
MW52-mm08	Groundwater[5]	Duplicate	MW-5	West of Furnaces		x	x	x	x	x											
MW53-mm08	Groundwater[5]	Duplicate	MW-18	Next to Little Vermillion River		x	x	x	x	x											
MW54-mm08	Groundwater[5]	Duplicate	MW-22	North Area		x	x	x	x	x											
MW55-mm08	Groundwater[5]	Duplicate	MW-28	East of Building 100		x	x	x	x	x											
MW56-mm08	Groundwater[5]	Duplicate	MW-30	North of Rolling Mill		x	x	x	x	x											
MW57R-mm08	Groundwater[5]	Rinsate	NA	sample group #1		x	x	x	x	x											
MW58R-mm08	Groundwater[5]	Rinsate	NA	sample group #2		x	x	x	x	x											
MW59R-mm08	Groundwater[5]	Rinsate	NA	sample group #3		x	x	x	x	x											
MW60R-mm08	Groundwater[5]	Rinsate	NA	sample group #4		x	x	x	x	x											
SW010-mm08	Surface Water[6]	Field	SW010	Ponded Water South of Building 100	NA	x	x	x	x	x	x				x		x				
SW011-mm08	Surface Water[6]	Field	SW011	Outflow from Main Industrial Area	NA	x	x	x	x	x	x				x						
SW012-mm08	Surface Water[6]	Field	SW012	Mouth-Abandoned Sewer Creek	NA	x	x	x	x	x	x				x						
SW013-mm08	Surface Water[6]	Field	SW013	Teminus-Abandoned Sewer Creek	NA	x	x	x	x	x	x				x						
SW014-mm08	Surface Water[6]	Field	SW014	North Area Standing Water	NA	x	x	x	x	x	x				x	x					
SW015-mm08	Surface Water[6]	Field	SW015	Outfall #1	NA	x	x	x	x	x	x				x						
SW016-mm08	Surface Water[6]	Field	SW016	Outfall #2	NA	x	x	x	x	x	x				x						
SW017-mm08	Surface Water[6]	Field	SW017	Outfall #3	NA	x	x	x	x	x	x				x						
SW018-mm008	Surface Water[6]	Field	SW018	Acid Reservoir	NA	x	x	x	x	x	x				x						
SW050-mm08	Surface Water[6]	Duplicate	SW014	North Area Standing Water	NA	x	x	x	x	x	x				x						

TABLE 2
PHASE II SAMPLING IDENTIFICATION INFORMATION FOR ALL MATRICES
Matthiessen and Hegeler Zinc Company Site, Operable Unit 2

Sample ID	Matrix	Sample Type	Station	Location	Depth (ft bgs)[1]	VOA	SVOA	Pest	PCB	Metals (Hg, CN)	Filtered Metals (Hg)	TCLP Metals	SPLP Metals	Asbestos	Total Hardness	Duplicate	MS/MSD	PCB field kit	Bioassessibility	Bioavailability	XRF Screening
QT001-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT002-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT003-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT004-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT005-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT006-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT007-mm08	Soil Boring	Trip Blank	NA	NA	NA	x															
QT008-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															
QT009-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															
QT010-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT011-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT012-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT013-mm08	Surface Water[6]	Trip Blank	NA	NA	NA	x															
QT014-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT015-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT016-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT017-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT018-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT019-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT020-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT021-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT022-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT023-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT024-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT025-mm08	Groundwater[5]	Trip Blank	NA	NA	NA	x															
QT026-mm08	Building Material[3,4]	Trip Blank	NA	NA	NA	x															

Notes:

All sampling locations with sample identification numbers are shown in Figures 2 through 5.

A	*Surface sample 0 to 2 ft bgs*
B	B: Subsurface sampling depth greater than 2 ft bgs
bgs	Below ground surface
BM	Building material
CN	Cyanide
ft	Feet
GW	Groundwater
Hg	Mercury
ID	Identification
mm	Two-digit month number
MS	Matrix spike
MSD	Matrix spike duplicate
MW	Monitoring well
NA	Not applicable
NE	Northeast
ns	Not screened; sample originally part of sample grid, but in-the-field decisions were made not to screen this location from July 28 to 30, 2008
NW	Northwest
OU	Operable Unit
PCB	Polychlorinated biphenyl
Pest	Pesticides
QT	Trip blank abbreviation for sample ID
R	Rinsate
SB	Soil boring
SPLP	Synthetic precipitation leaching procedure
SS	Soil sample
SVOA	Semivolatile organic analysis
TBD	To be determined
TCLP	Toxicity charateristic leaching procedure
VOA	Volatile organic analysis
X	Sample collected for XRF calibration study; analyzed by Contract Laboratory Program laboratory
XRF	X-ray fluorescence

1	All B depths - a specific 2-ft interval will be determined in the field based on requirements in Section 5.2 of the field sampling plan
2	Poly_1, Poly_2, etc., refer to polygon 1, polygon 2, etc., made up in gridded sampling areas throughout OU2 for the XRF screening and suface soil sampling campaign
3	For building samples, only organic samples (wood) or samples with a stained appearance will also be analyzed for VOCs.
4	Sample ID for building materials must include substrate/material composition in sample identification name as in Table 9 of the field sampling plan (determination to be made in the field).
5	Samples will be collected from all 36 monitoring wells for analysis for VOCs, SVOCs, pesticides, PCBs, metals (including Hg), and cyanide during the initial four quarterly sampling events. After the initial quarterly sampling events, analytical data will be evaluated to determined the chemicals of interest/analyte groups to be sampled for in each well.
6	Surface water samples will be collected in June and October 2008.

FIGURES

(Five Pages)

FIGURE 1: SITE LOCATION MAP

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

FIGURE 1
SITE LOCATION MAP

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

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FIGURE 2: PROPOSED SOIL BORING LOCATION MAP

NORTH AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB421	21
SB422	22
SB423	23
SB424	24
SB425	25
SB426	26
SB427	27
SB428	28
SB429	29
SB430	30

NEAR BLDG 100 (PCB AREA)	
SOIL BORING ID	MAP ID# & SYMBOL
SB401	1
SB402	2
SB403	3
SB404	4
SB405	5
SB406	6
SB407	7
SB408	8
SB409	9
SB410	10

NORTHWEST OF ROLLING MILL	
SB411	11
SB412	12
SB413	13
SB414	14
SB415	15
SB416	16
SB417	17
SB418	18
SB419	19
SB420	20

NORTHEAST PERIPHERY AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB441	41
SB442	42
SB443	43
SB444	44
SB445	45
SB446	46
SB447	47
SB448	48
SB449	49
SB450	50

INSIDE ROLLING MILL	
SOIL BORING ID	MAP ID# & SYMBOL
SB431	31
SB432	32
SB433	33
SB434	34
SB435	35
SB436	36
SB437	37
SB438	38
SB439	39
SB440	40

MAIN PLANT AREA	
SOIL BORING ID	MAP ID# & SYMBOL
SB451	51
SB452	52
SB453	53
SB454	54
SB455	55
SB456	56
SB457	57
SB458	58
SB459	59
SB460	60

LEGEND:

OPERABLE UNIT OU2 SITE BOUNDARY

ROLLING MILL — HISTORIC STRUCTURE (APPROXIMATE LOCATION) (NOT ALL HISTORIC STRUCTURES ARE OUTLINED)

GRAPHIC SCALE

(IN FEET)
1 INCH = 150 FEET

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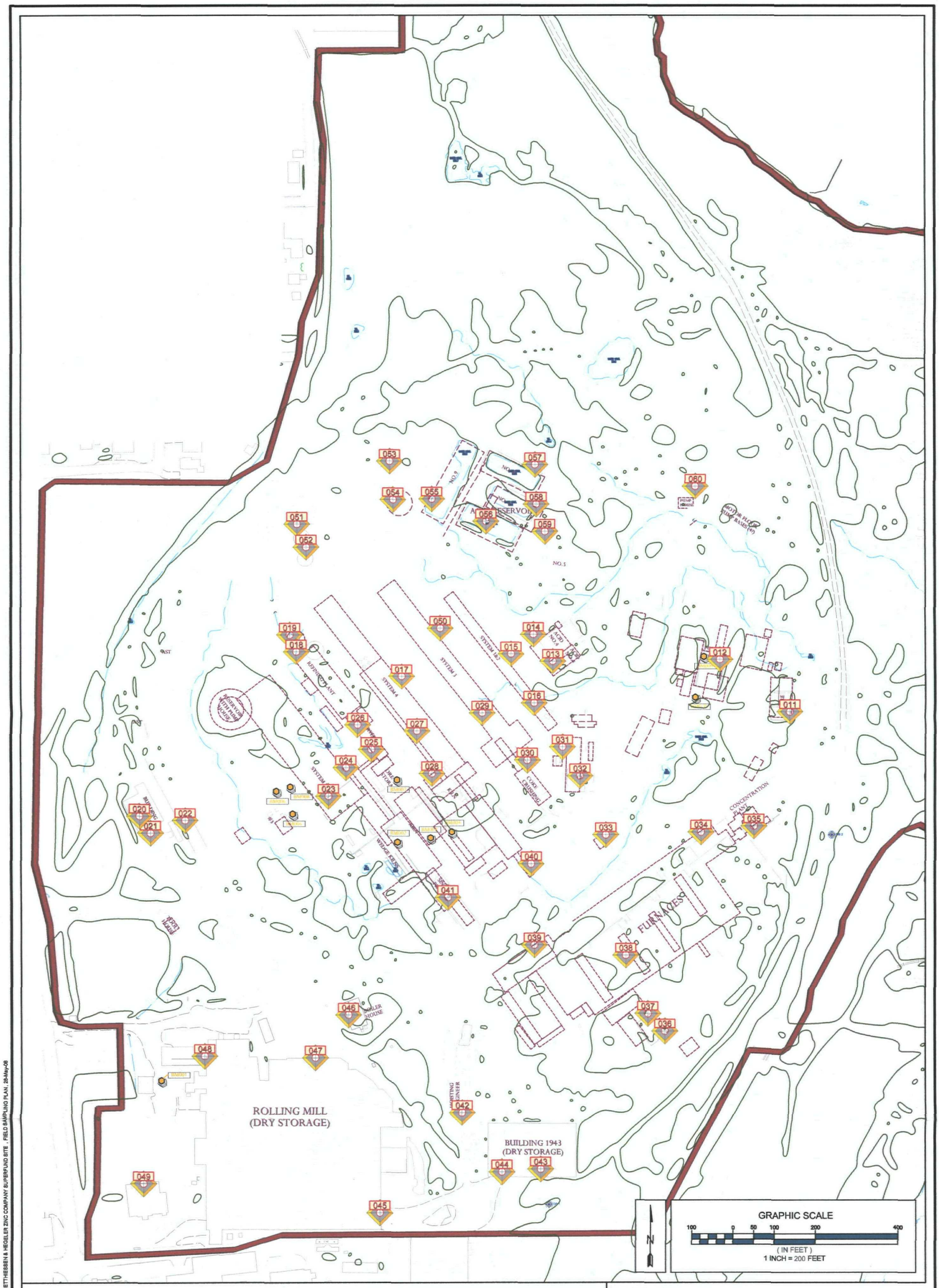
MATTHIESSEN & HEGELER ZINC COMPANY SITE
STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS
EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

FIELD SAMPLING PLAN
FIGURE 2
PROPOSED SOIL BORING LOCATION MAP

DOUGLAS B. GRANT, P.G & DR. JENNIFER LAWSON-KOBRIS, PhD. - MATTHIESSEN & HEGELER ZINC COMPANY SURROUND RI/FS - FIELD SAMPLING PLAN_30-Nov-08

FIGURE 3: PROPOSED BUILDING SAMPLE LOCATION MAP



LEGEND:

- PROPOSED BUILDING MATERIAL SAMPLE LOCATION (60) BM-011-08 TO BM-060-08
- PREVIOUS BUILDING MATERIAL SAMPLE LOCATION (10) BM-001 TO BM-010
- HISTORIC STRUCTURE (APPROXIMATE LOCATION) (NOT ALL HISTORIC STRUCTURES ARE IDENTIFIED)
- OPERABLE UNIT OU2 SITE BOUNDARY

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

FIGURE 3

PROPOSED BUILDING SAMPLE LOCATION MAP

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

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DOUGLAS B. GRANT, PG & DR. JENNIFER LAWSON-KNOEPFLE, PHD. \METTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE . FIELD SAMPLING PLAN, 28-May-08

FIGURE 4: PROPOSED MONITORING WELL AND PIEZOMETER LOCATION MAP

MW 22
MW 35
DB MW 35
MW 20
MW 23
ACID RESERVOIRS
MW 21
P1
DB P1
MW 24
MW 25
DB MW 25
P4
P5
MW 19
P6
MW 27
MW 28
P2
MW 26
MW 31
FURNACES
MW 30
P3
MW 29
MW 32
ROLLING MILL
(DRY STORAGE)
BUILDING 1943
(DRY STORAGE)
MW 33
MW 34

GRAPHIC SCALE
125 0 125 250 500
(IN FEET)
1 INCH = 250 FEET

N

LEGEND:

- MW 34 — PROPOSED MONITORING WELL LOCATION (17)
- P6 — PROPOSED LOCATION OF 2-INCH DIAMETER PIEZOMETER (6)
- DB MW 35 — PROPOSED DEEP BORING (3)*
- EXISTING GROUNDWATER MONITORING WELL (17)
- EXISTING GROUNDWATER MONITORING WELL CLUSTER - DEEP AND SHALLOW SCREENS (1 GROUP OF 2 WELLS)

NOTES:

* BACKFILLED PRIOR TO INSTALLATION OF PIEZOMETER P1 AND MONITORING WELLS MW 25 AND MW 35

MATTHIESSEN & HEGELER ZINC COMPANY SITE

FIELD SAMPLING PLAN

STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS

FIGURE 4

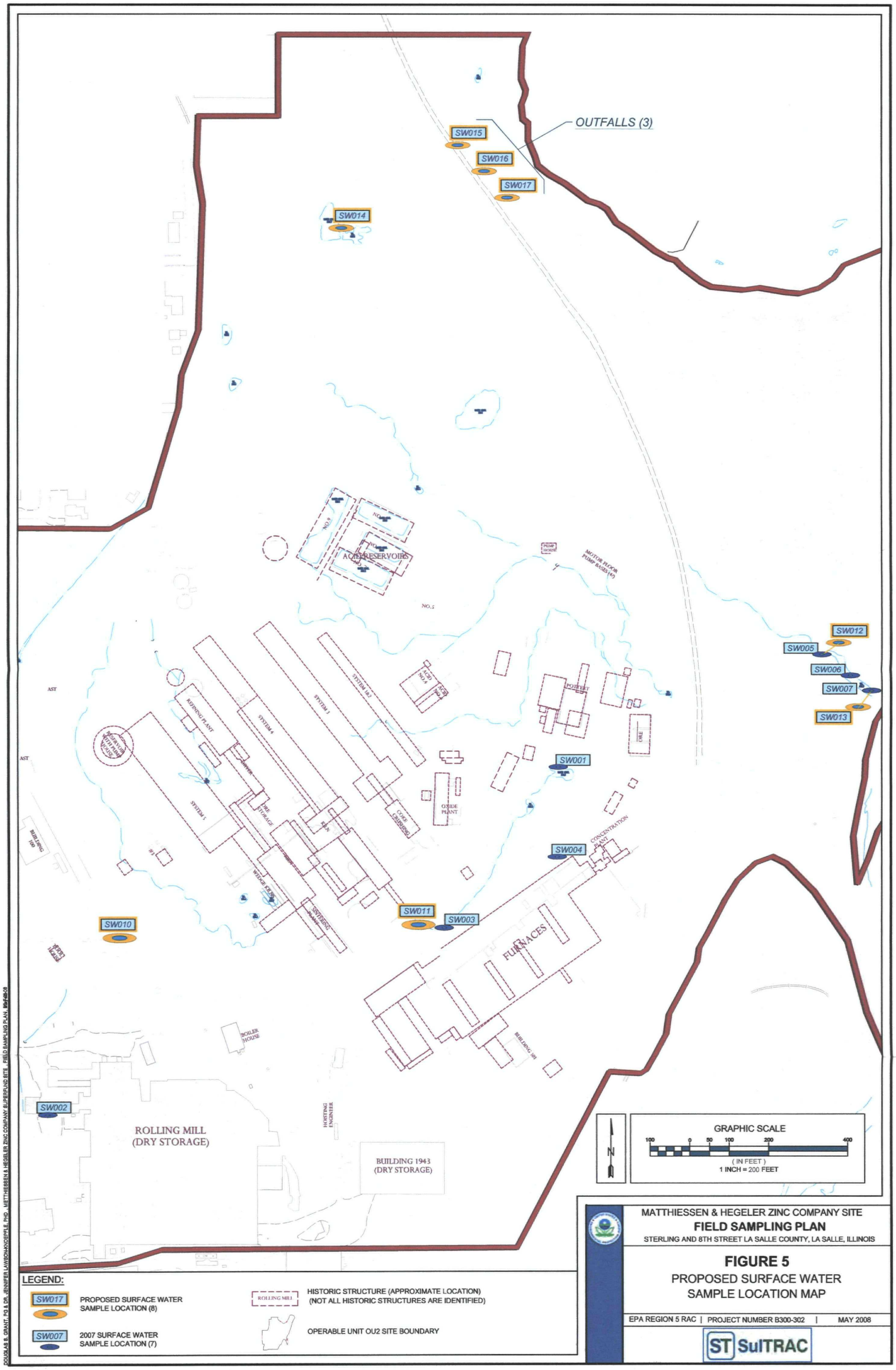
EXISTING AND PROPOSED MONITORING WELL AND PIEZOMETER LOCATION MAP

EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008

ST SulTRAC

DOUGLAS B. GRANT, PG & DR. JENNIFER LAWBOHKNOBPLE, PHD , MATTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE , FIELD SAMPLING PLAN, 30-May-08

FIGURE 5: PROPOSED SURFACE WATER SAMPLE LOCATION MAP


OUTFALLS (3)
SW015
SW016
SW017
SW014
SW012
SW005
SW006
SW007
SW013
SW001
SW004
SW011
SW003
SW010
SW002
ACID RESERVOIRS
NO. 5
NO. 4
NO. 3
NO. 2
NO. 1
PUMP HOUSE
MOTOR FLOOR PUMP BASES (4?)
POTTERY
ACID NO. 4
ACID NO. 3
SYSTEM 1&2
SYSTEM 3
SYSTEM 4
SYSTEM 5
REFINING PLANT
DRYER
ORE BIN
ORE
OXIDE PLANT
COKE CRUSHING
ORE STORAGE
KILN
WEDGE KILN
SINTERING PLANT
CONCENTRATION PLANT
FURNACES
BUILDING 101
BUILDING 100
RESERVOIR WITH PUMP HOUSE
AST
AST
BOILER HOUSE
HOISTING ENGINEER
ROLLING MILL
(DRY STORAGE)
BUILDING 1943
(DRY STORAGE)
N
GRAPHIC SCALE
100 0 50 100 200 400
(IN FEET)
1 INCH = 200 FEET
LEGEND:
SW017
PROPOSED SURFACE WATER SAMPLE LOCATION (8)
SW007
2007 SURFACE WATER SAMPLE LOCATION (7)
ROLLING MILL
HISTORIC STRUCTURE (APPROXIMATE LOCATION) (NOT ALL HISTORIC STRUCTURES ARE IDENTIFIED)
OPERABLE UNIT OU2 SITE BOUNDARY
MATTHIESSEN & HEGELER ZINC COMPANY SITE
FIELD SAMPLING PLAN
STERLING AND 8TH STREET LA SALLE COUNTY, LA SALLE, ILLINOIS
FIGURE 5
PROPOSED SURFACE WATER SAMPLE LOCATION MAP
EPA REGION 5 RAC | PROJECT NUMBER B300-302 | MAY 2008
ST SulTRAC
DOUGLAS B. GRANT, PG & DR. JENNIFER LAWSON-KNOEPFLE, PHD, MATTHIESSEN & HEGELER ZINC COMPANY SUPERFUND SITE - FIELD SAMPLING PLAN, 05/28/08